september 1959

nlgi spokesman

journal of the national lubricating grease institute

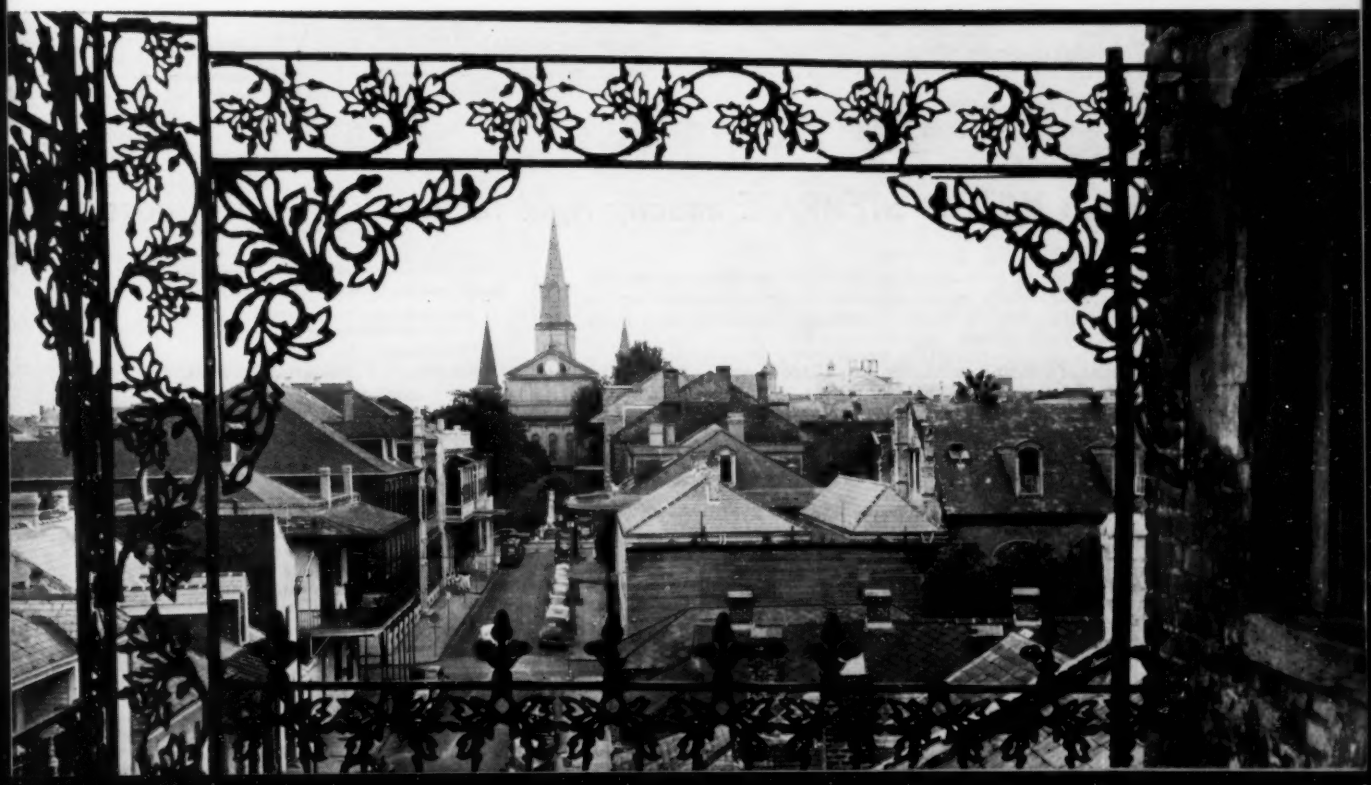
Viscoelastic Behavior of Greases

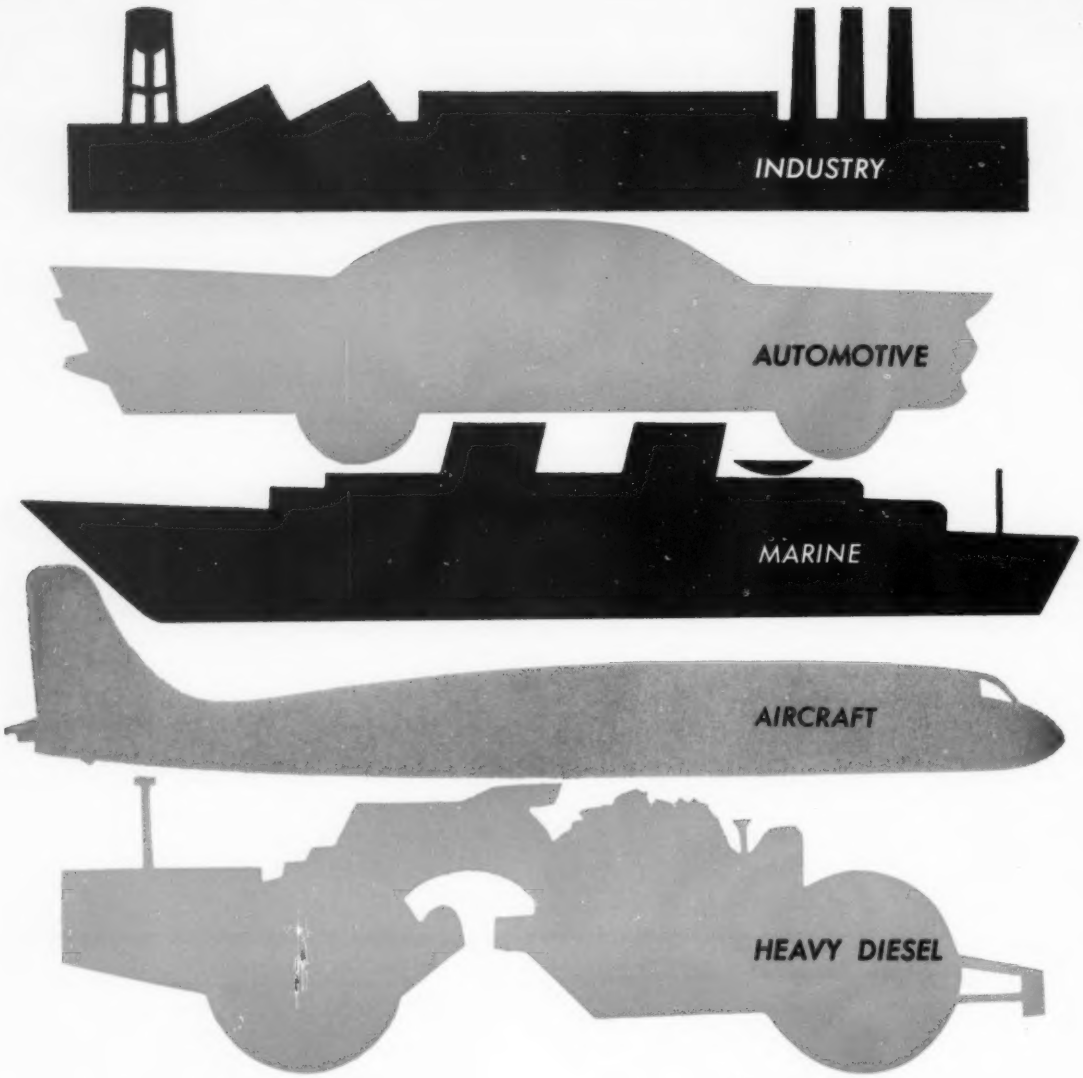
By E. O. FORSTER and J. J. KOLFENBACH

Previews—NLGI 27th Annual Meeting

Type A, and Type A, Suffix A, Automatic Transmission Fluids

By W. H. KITE, JR.





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THE COVER

BALCONIES of iron lacework in old New Orleans are just one of the many attractions the ladies will see in their tours of the Crescent City, while a carefully thought out program for all segments of the industry will make the 27th Annual Meeting an outstanding attraction for marketer and technical expert alike. Wives are cordially invited and there are a number of activities during the October 25-28 session which the ladies may enjoy . . . receptions, "get-acquainted" coffees, tours, lunches, a social hour and banquet, and plenty of free time for that all-important shopping!

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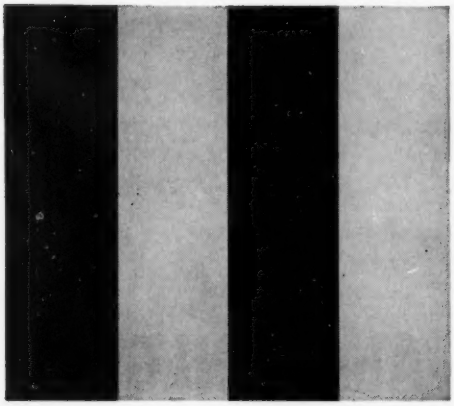


NLGI PRESIDENT'S PAGE

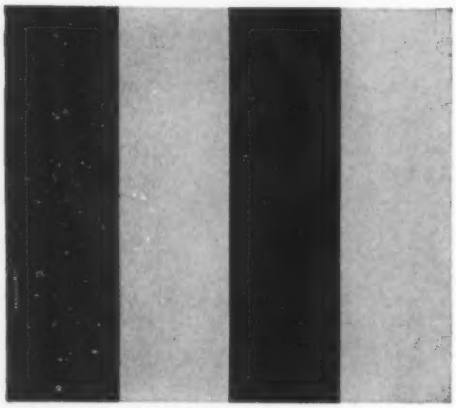
By F. E. ROSENSTIEHL, *President*

1959 Annual Meeting

Ever since the annual meeting last year, your Program Committee has been actively engaged in setting up an outstanding program of interest to all segments of our industry for this year's meeting. A well-balanced selection of papers, covering subjects of vital interest to all phases of our industry will be presented, these covering the Chassis Lubricant Market—including status of non-lubricated, non-metallic bearings. Foreign grease manufacturers' facilities will be covered in another paper, as will new developments in grease composition and containers. Entire separate sessions will be devoted to spotlighting manufacturers' production equipment and dispensing problems. As usual a full session will be devoted to Technical Committee activities.

Taking into consideration the calibre and diversity of the subjects to be discussed makes it imperative that anyone having a connection with any facet of the grease industry make every effort to attend the annual meeting. Added to this is the romantic interest of New Orleans, where the meeting will be held, and the fact that a separate committee is arranging items of interest to all women in attendance should make this one of the best attended and a most outstanding meeting.

Make your plans NOW to attend. ■



News About NLGI

Changes in NLGI Representatives Noted

The following changes have been noted among the member firms of NLGI, concerning the men selected to represent each company as company and technical representative:

Royal Lubricants has named J. C. Mosteller, vice president, as company representative of this Active member firm.

Lithium Corporation of America has appointed G. L. Brown as its technical representative. LCA is an Associate member firm.

Jones & Laughlin Steel, an Associate member, has designated J. E. Morris, general manager, container division, in the dual capacity of company and technical representative to NLGI.

Harchem Division, Wallace & Tierman, has named H. M. Abbott and Dr. W. P. Brown as the company and technical representatives from this Associate member.

Sinclair Refining has listed F. W. Minor as company representative, replacing the late Mr. A. S. Randak. George Entwistle, chief chemist, and for several years head of the NLGI SPOKESMAN review committee, has been made the NLGI technical representative for this Active member firm.

Western Oil, a Canadian Active, has named Harold Birch, grease maker, as company representative, while Vernon L. Hoover will serve as technical committee representative.

Enjay, an NLGI Associate member, appointed Warren C. Wilson, product manager, paramins division, as both company and technical representative.

Rieke Metal Products has named Raymond F. Ouer, general sales manager, as company representative of this Associate member firm.

Sumner Sollitt company, an Associate NLGI member, has named as technical representative R. N. Mullin, director of petroleum industry activities.

Lubrizol, Associate member firm, has named J. B. Irwin as company representative.

Name Change for NLGI Member

Texaco Canada Limited is now the name of the NLGI Active member in Canada formerly known as McColl-Frontenac Oil Company, Ltd. The address is still 1425 Mountain St., Montreal 25, Quebec, Canada. R. N. Smith is both company and technical representative to NLGI.

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WHEEL BEARING MANUAL— "Recommended Practices for Lubricating Automotive Front Wheel Bearings." More than 150,000 copies of this booklet have been distributed throughout the world. Twenty cents a copy (NLGI member price) with quantity discounts — company imprint arranged.

REPRINTS — From the NLGI SPOKESMAN are available at low cost. Page forms are left standing for three months, company imprint or advertising arranged.

NLGI MOVIE — "Grease, the Magic Film," a 16-mm sound movie in color running about 25 minutes, now released. First print $300, second and subsequent orders $200 each (non members add $100 to each price bracket).

VOLUME XXII

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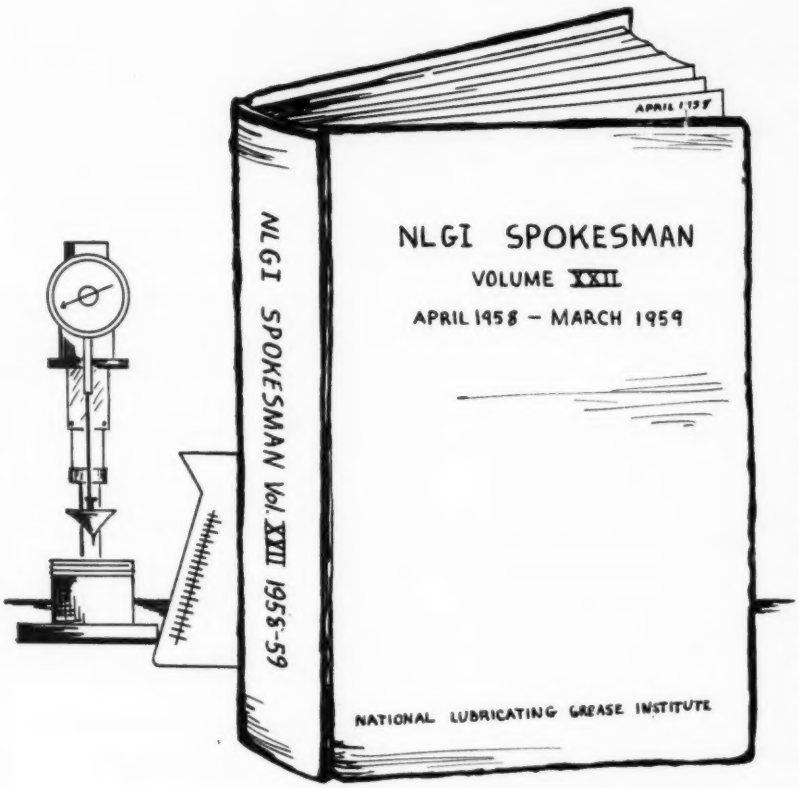

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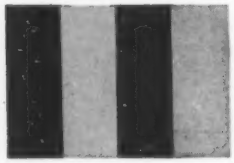

Future Meetings

SEPTEMBER, 1959

14 NLGI Board of Directors Meeting, Hotel Roosevelt, New York City.

16-17 Ohio Petroleum Marketers Association, Fall Conference and Golf Tournament, Sheraton-Cleveland Hotel and Lakewood Country Club, Cleveland, Ohio.

16-18 NPA Annual Meeting, Hotel Traymore, Atlantic City, N.J.

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27-29 IOCA 12th Annual Meeting, Pick-Congress Hotel, Chicago, Ill.

OCTOBER, 1959

2-3 Association of Desk and Derrick Clubs of North America, 8th Annual Convention, Hilton Hotel, San Antonio, Tex.

11-15 ASTM Committee D-2 Meeting, Sheraton - Palace Hotel, San Francisco.

12-13 Petroleum Packaging Committee Meeting, Port Arthur, Texas

20-22 ASLE and ASME Joint Lubrication Conference, Sheraton-McAlpin Hotel, New York City.

26-28 NLGI Annual Meeting, Roosevelt Hotel, New Orleans, La.

28-30 Society of Automotive Engineers, National Fuels and Lubricants, La Salle Hotel, Chicago.

NOVEMBER, 1959

9-11 API, 39th Annual Meeting, Conrad Hilton, Palmer House and Congress Hotels, Chicago.

10-12 API, Marketing Division, Congress Hotel, Chicago.

DECEMBER, 1959

11-15 Society of Automotive Engineers, Meeting, Sheraton-Cadillac and Statler Hotels, Detroit.

JANUARY, 1960

31-Feb. 5 ASTM, Committee D-2 Meeting, Statler Hotel, Detroit.

FEBRUARY, 1960

25-26 API Division of Marketing, Lubrication Committee Meeting, Sheraton - Cadillac Hotel, Detroit.

OCTOBER, 1960

31-Nov. 1 NLGI Annual Meeting, Edgewater Beach Hotel, Chicago.

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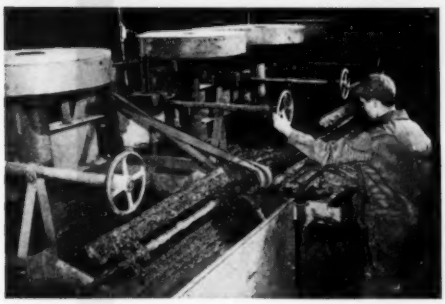

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All-out motorist promotion by the Frontier Refining Company brings in new customers, dramatically increases service station traffic

▲ Greeley motorists were made vividly aware of "Moly" grease by covered wagon with provocative banners. Wagon toured streets day after day, made frequent stops at Greeley's four Frontier stations.

◀ Typical Frontier station in Greeley is operated by Robert Innis at 1028 Ninth Street. Station, in residential district off main through highways, depends on repeat business of satisfied local residents.

"MOLY" GREASE HIT GREELEY, COLO.

TYPICAL STATION MULTIPLIED GREASE SALES EIGHT TIMES; GAS SALES UP 10%

Though abnormally bad spring weather dampened sales for most service stations in Greeley, the Frontier Refining Company's outlets showed phenomenal increases. Frontier, who recently adopted *Molysulfide®* chassis grease for its service stations, had launched an all-out motorist promotion of "Moly" grease to increase service station traffic.

Results at Bob Innis' Frontier station in Greeley were typical of the response—and further proof that "Moly" grease excites the imagination and interest of both motorists and dealers. Innis reports:

> "I averaged 12 to 15 grease jobs a month before Frontier introduced 'Moly-Lith' (Frontier's brand name for its 'Moly' grease.) After putting in 'Moly-Lith', my grease sales jumped to 98 in the first 28 days. I estimate that 35 to 40% of these sales were to new customers, and I honestly believe they were attracted to my station from the 'Moly-Lith' promotion alone.
>
> "My gasoline sales increased 10% in spite of the bad weather. I can also see an increase in my sales of TBA.
>
> "Frontier 'Moly-Lith' grease has put the car on the rack, and, by my being able to get under the hood, I'm selling more fan belts, batteries, universal joints and oil filters."

TREND IS TO "MOLY" GREASE IN STATIONS

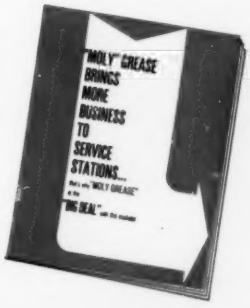
Oil marketers in growing numbers are building service station sales with "Moly" grease. The reason is simple. Its advantages are easy to demonstrate, easy to understand—*and, above all, easy for the customer to experience*. Motorists come back for it . . . ask for it . . . tell their friends to ask for it. And, as Frontier's experience shows, this increased traffic increases sales of everything the station sells.

Investigate the merchandising potential of "Moly" grease for your service stations. Climax Molybdenum Company, a Division of American Metal Climax, Inc., 500 Fifth Avenue, New York 36, N. Y.

Triple-play promotion involved coordinated effort by Frontier sales organization, advertising department and station operators. L. to R.: Dale Wright, Frontier Advertising Director; Bob Innis, local operator; Alan Gardner, Frontier's Manager of Lubricant Sales.

Special month-long promotion included big newspaper ads (in color) twice a week, 12 radio spots a day over two stations. Meetings were held with groups of truckers and farmers. Posters were supplied to stations, as were steering wheel tags to put on cars after grease job.

Write for your copy of " 'Moly' Grease Brings More Business to Service Stations". This case history tells how another major oil company used "Moly" grease to promote sales.



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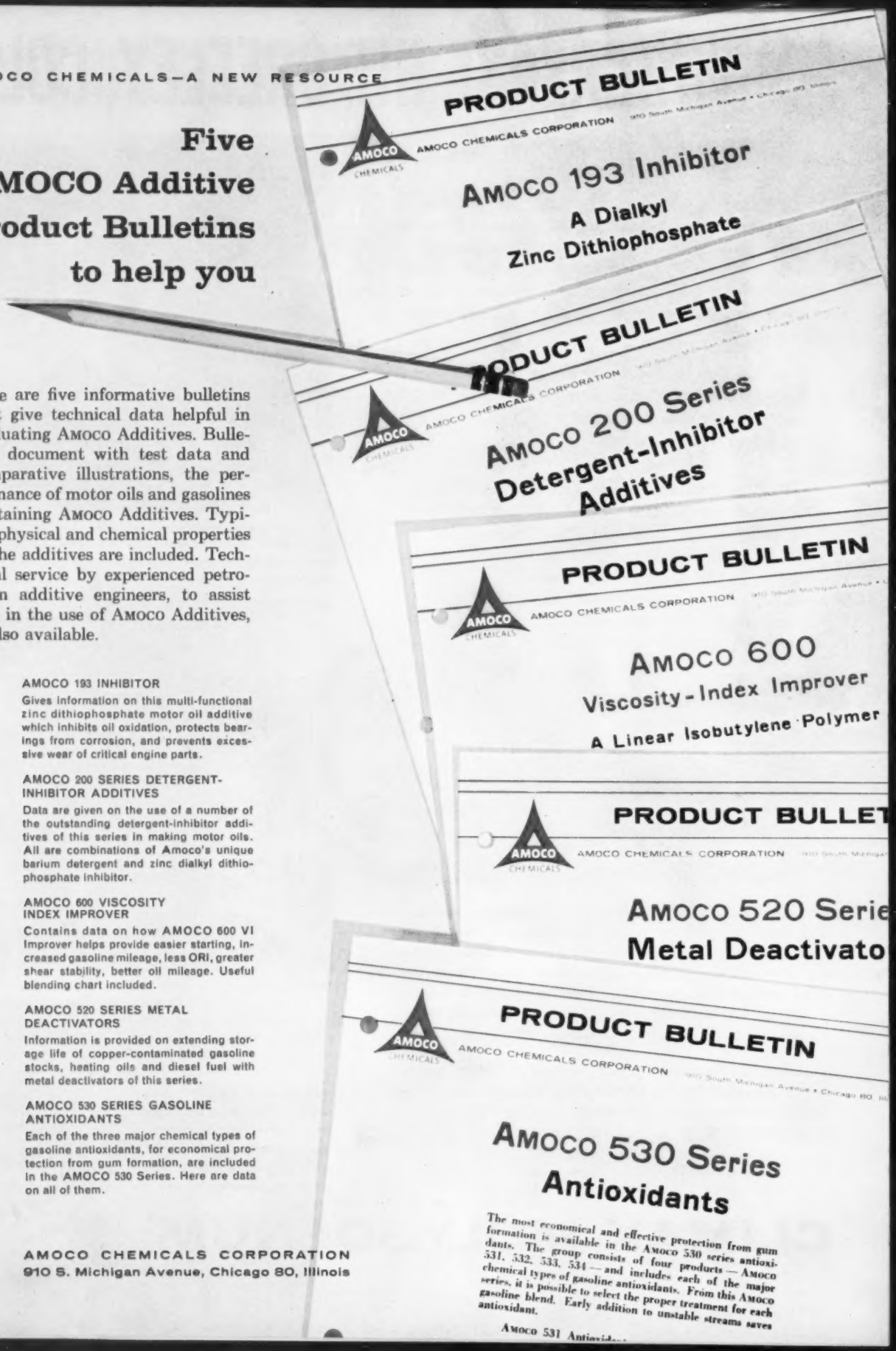
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Viscoelastic Behavior of Greases

By: E. O. Forster
J. J. Kolfenbach
Esso Research and Engineering Co.

Presented at the ASLE ASME Lubrication Conference, Los Angeles, October, 1958

1. Introduction

The mechanical properties of greases are usually examined either under steady state conditions at relatively high shearing stresses or under static conditions involving very small shearing stresses. The steady state conditions, such as encountered in the capillary pressure viscometer, are selected in the belief that they represent the conditions under which the greases will have to operate. The static conditions such as those encountered in the grease penetrometer are usually employed as specification tests to permit rapid comparisons between materials having the same chemical composition. Relatively few investigations have been made of the behavior of greases which reflect the time dependent behavior of these materials. Vinogradov and Klimov (1) examined calcium soap greases with a torsional elastometer and found that under low shear stresses, greases behaved like an elastic solid body. At higher shear stresses they found that these elastic deformations were accompanied by irreversible plastic deformations and that description of the mechanical behavior of greases required a knowledge of the shear modulus, as well as the viscosity, as a function of time. Carver and Van Wazer (2) and Goldberg and Sandvik (3) studied aluminum gels using a concentric cylinder type viscometer which could be made to rotate suddenly at a constant speed or to oscillate mechanically at frequencies ranging from 10 to 6000 cycles per second. Both research teams found that the aluminum soap gels had elastic as well as viscous properties and resembled linear amorphous polymers in their mechanical behavior. Hutton and Matthews (4) investigated simple sodium and lithium soap greases in a parallel plate plastometer under compression and confirmed again that these greases had elastic as well as viscous properties.

In the present paper, the behavior of a mixed soap and a complex soap grease is described under cyclic shear stresses resulting in cyclic deformations. The results of this investigation support earlier findings that greases are viscoelastic bodies and that their response to shear stresses is very time dependent. From the study of the frequency dependence of these materials under a wide range of frequencies, the distribution of relaxation times has been deduced. Evidence has been obtained of the existence of three different types of relaxation mechanisms which are believed to correspond to different types of displacement that can take place within greases. These results are interpreted as providing further evidence of the structure of greases postulated by Hutton (4), Forster and Kolfenbach (5)—namely, that greases are made up of a three-dimensional soap fiber network in which the oil is immobilized.

2. Experimental Techniques

The mechanical properties of viscoelastic materials can be studied with three types of rheological instruments according to the specific interest of the investigation. If only the flow properties of a viscoelastic material are to be studied, viscometers such as the Couette or Pochettino coaxial cylinder type can be used. On the other hand, if one is interested in viscoelastic effects that occur in times longer than about 10 sec, one can use static (transient) experiments in which either the strain is observed under constant stress (creep measurements) or the stress is observed under constant strain (relaxation measurements) using instruments such as the elastometer or plastometer. Finally, if investigations are to be made of the viscoelastic effects occurring in times shorter than ten secs, dynamic measurements have to be made. Naturally there exists among these methods a certain amount of overlap with regard to the time scale.

The majority of dynamic tests are based upon vibrating systems in which stress and strain varies sinusoidally with time. The time scale involved ranges from about 10^{-8} sec up to 10^{4} sec (6). The dynamic measurements can be grouped into three classes based upon the ratio of wave-lengths of the elastic waves generated to the dimensions of the sample. If these dimensions are small compared to the wavelength, one uses the method of vibration with additional mass. In order to set up vibrations, an additional mass is fixed to the sample and the system permitted to vibrate freely or forced to vibrate at certain frequencies. If the dimensions of the sample are of the same order of magnitude as the wave lengths, one uses vibrations without additional mass. In this case, standing waves, which are dominated by the mass of the sample, are built up. If the dimensions of the sample are very large compared with the wavelengths, elastic waves are propagated through the medium and one employs wave propagation methods.

In the work of Vinogradov (1), static experiments were conducted to examine the stress relaxation of a grease. Carver (2) in his study of aluminum soap gels used a Couette type viscometer to study the flow properties, and Goldberg (3) used a similar arrangement to which he added a forced oscillatory motion, thus performing an experiment of the forced vibration type with additional mass. Hutton (4) subjected his grease to compression and performed essentially creep measurements. The present investigation employed forced vibrations with additional mass similar to that of Goldberg (3) but unlike Goldberg, the imposed frequencies

were very low compared to the resonance frequency of the sample. This non-resonance method was selected because it appears to be the most appropriate method for measuring large energy losses in viscoelastic materials.

A. *Theory*

Before the difference between the resonance method and the non-resonance method can be appreciated, it appears advisable to discuss briefly the theoretical background of the dynamic test method. A viscoelastic material which is subjected to sinusoidal shearing stresses of maximum amplitude τ_0 will reach a balance of stresses and deformations in a few cycles. At any particular time, t, the stress can be written as

$$\tau = \tau_0 \sin \omega t \qquad [1]$$

where ω is angular frequency of vibration. The corresponding strains will also vary sinusoidally with the same frequency with an amplitude γ_0 and with a certain phase difference δ. At any time, t, the shear strain will be given by the equation

$$\gamma = \gamma_0 \sin(\omega t - \delta) \qquad [2]$$

Thus, it can be seen that the strain will lag behind the stress. The phase angle that exists between the two is called the loss angle δ. The ratio between stress and strain amplitudes is called the absolute dynamic modulus, $|G|$,

$$|G| = \tau_0/\gamma_0 \qquad [3]$$

The absolute dynamic modulus together with the loss angle are sufficient to describe the dynamic behavior of a viscoelastic material.

For reasons of convenience, one introduces two other characteristic functions for the description of vibration experiments. The first one, G_1 is called the storage modulus or modulus of rigidity. The second one, G_2, is called the loss modulus. This loss modulus is directly related to the dynamic viscosity η' by the equation

$$\eta' = \frac{G_2}{\omega} \qquad [4]$$

The stress-strain relationship can then be expressed in the following manner:

$$\tau = \gamma_0[G_1(\omega) \sin \omega t + G_2(\omega) \cos \omega t] \qquad [5]$$

The first term in this expression is the component of the stress in phase with strain and determines the elastic reactive forces. The second term represents the part of the stress which is 90° out of phase with the strain and represents the dissipative forces. The relationships between the absolute modulus, the loss angle and the storage and loss moduli are given in the following equations

$$G_1 = |G| \cos \delta;\ G_2 = |G| \sin \delta;\ \tan \delta = G_2/G_1 \qquad [6]$$

The meaning of the loss modulus can be best illustrated by the calculation of the dissipation of energy due to viscoelastic losses in the material. The energy dissipated during one cycle is

$$W = \int \tau d\gamma = \pi G_2(\omega)\, {}_0{}^2 = \pi \tau_0 \gamma_0 \sin \delta \qquad [7]$$

while the total energy expanded during one cycle, E, is given by

$$E = \pi \tau_0 \gamma_0 \qquad [8]$$

The storage and loss moduli are sometimes expressed also as a single complex quantity, the complex shear modulus, $G^* = G_1 + iG_2$, where the moduli are the real and imaginary parts of the complex modulus.

With this background in mind, one can now appreciate the difference that exists between the method of forced vibrations operating at a frequency close to the resonance frequency of the material and the non-resonance case. In the resonance case, one can handle only conditions under which tan δ is much smaller than 1. In this case the moduli vary only slowly with frequency and one measures the amplitude as a function of the frequency ω in the neighborhood of the resonance frequency ω_0. In the non-resonance case the tangent of the loss angle is not restricted to being much smaller than 1. In this case, force and deformation are compared with respect to amplitude and phase. As a matter of fact one will find in most text books dealing with this subject, that the discussion of vibration measurement including definitions of storage and loss modulus are based upon non-resonance forced vibrations and the resonance case is only considered as a special limited application.

B. *Apparatus*

The vibration tester used in this investigation was developed by W. Philippoff[7] and has not been described in detail elsewhere. Basically, the instrument consists of two concentric cylinders, the outer one referred to as the cup and the inner one is called the plunger. With this setup, the material is pumped by movement of the inner cylinder. The plunger is mounted in a frame which is connected through a dynamometer to a driving cam which imparts sinusoidal motion in the direction of the long axis of the plunger. The cup is solidly clamped to the base of the machine. Between the cam and the frame, as well as between the cup and the frame, strain gages are mounted to permit the measurement of the stress and the movement of the frame. Two linear-variable transformers were used in conjunction with these strain gages. The output of these transformers, properly amplified and rectified, was applied to the horizontal and vertical plates of an oscilloscope. The resulting stress-strain ellipse traced on the screen of the oscilloscope was photographed and the picture used to obtain the necessary experimental information. The details of the analysis of this ellipse and the method used to calculate the moduli are shown in Appendix I. When frequencies below 10^{-3} cycles per sec were used, the output of the strain gages was recorded on a two-pen recorder rather than an oscilloscope because the long exposures necessary to record a complete cycle produced poor pictures.

The range of forces and deformations that can be measured with this equipment varies from a few grams to 10 kilograms force and from 10 to 1000 microns double amplitude of deformation. The frequencies covered with this instrument range from 10^{-4} to 10 cycles per second and the test temperature ranges from −17°C to 50°C.

C. *Procedure*

Two sets of plungers were used with two different cups, thus providing clearances ranging from 2/32 to 8/32 inches. The plunger and cup sets were calibrated with a National Bureau of Standards oil. From this calibration, the instrument constants were obtained. These calibration values are shown in Table 1 together with the values that were obtained from theoretical calculations based on the geometry of the cup and plunger arrangement. The details of these calculations are shown in Appendix I.

TABLE 1
Calibration Values

	Constants in dyn × microns cm^2 kg	
Gap, Inches	*Calculated*	*Experimental*
2/32	1.035×10^4	1.185×10^4
3/32	3.26×10^4	3.24×10^4
7/32	4.98×10^5	5.07×10^5
8/32	7.15×10^5	6.31×10^5

It can be seen from this comparison that there exists good agreement between these two sets of values.

Once the instrument was calibrated, the grease samples were carefully packed into the cup to avoid any entrapment of air and any unnecessary working. The sample was then brought up to the desired temperature and was ready for testing.

D. *Materials*

Two greases were used in this investigation. Grease A was a mixed calcium-lithium soap grease of NLGI grade 2 and grease B was a complex calcium soap grease of NLGI grade 1. Both greases contained naphthenic type base oils with grease A containing a slightly more viscous oil than grease B. The composition and physical properties of these greases are shown in Table 2.

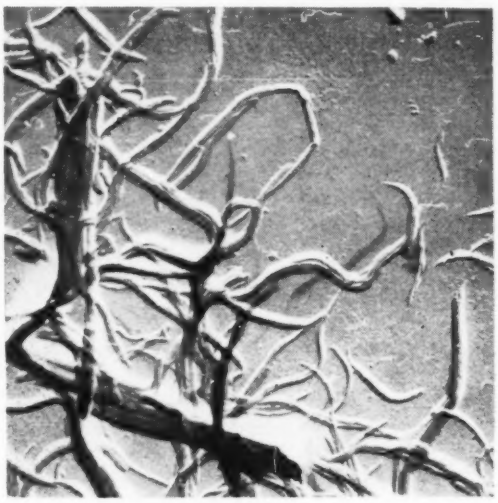
TABLE 2
Composition and Physical Properties of Test Greases

Composition	*Grease A*	*Grease B*
Soap Type	*Lithium/ Calcium*	*Complex Calcium*
% Soap content	12	22
Base oil viscosity, SSU at 210°F	70	60
V.I. of base oil	35	35
Physical Properties		
Penetration, mm/10 at 77°F		
Unworked	275	274
Worked 60 strokes	280	310
Worked 10,000 strokes	320	333
Unworked at 210°F	350	300

The main difference between these two greases existed in the shape and size of their constituent soap fibers as shown in Figure 1. From the electron micrographs, it can be seen that grease A has fibers with a length to diameter ratio of about 1000 to 1 or more, while in grease B this ratio is of the order of 10 to 1. These micrographs were made from the samples of the two greases after homogenization. It should be kept in mind that all the following results were obtained from these homogenized samples.

3. Experimental Results

Both greases were examined in the vibration tester to determine their dependence on frequency, amplitude, temperature, and the geometry of the testing equipment.



Grease A

Grease B

FIGURE 1, comparison of soap structure, 10,000×.

A. *Reproducibility of Sample Preparation*

Before proceeding with the above mentioned measurements, it was necessary to determine the reproducibility of the measurements as affected by sample preparation. Fresh samples of greases A and B were examined at several frequencies and the values so obtained for G_1 and G_2 were subjected to a statistical analysis. From this analysis, it was concluded that the reproducibility of samples was very good and that one could obtain the values of the moduli with an accuracy of $\pm 5\%$ at 90% confidence level.

When measurements are repeated after a whole sequence of tests had been run with the same sample, significant changes outside the experimental error were observed. This behavior of the greases is a reflection of their sensitivity to working and is illustrated in Table 3.

TABLE 3

Effect of Working on Rigidity and Viscosity of Test Greases
(Frequency $f = 0.034$ c/s, 25°C)

	Grease A			*Grease B*		
Run No.	G_1'	G_2[a]	η'[b]	G_1[a]	G_2[a]	η'[b]
1	3.81	1.88	8.85	2.54	1.61	7.52
2	4.14	1.62	7.60	2.03	1.23	5.75

[a] In 10^3 dyn/cm^2

[b] $\eta' = G_2/2\pi f$ in 10^3 poises

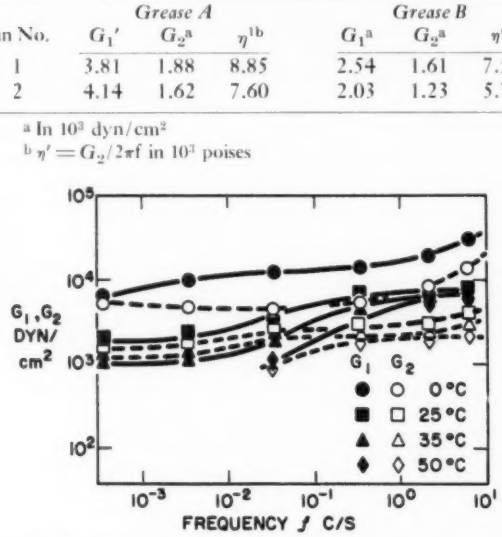


FIGURE 2, frequency dependence of grease A.

There was a difference of two days between the first and the second run during which period the grease sample had been subjected to a complete frequency and amplitude analysis.

These observations point out the need for careful control of the conditions when testing greases, since the properties of these materials depend on their previous history. Such behavior had been noticed earlier by Singleterry and Stone.[8]

B. *Frequency Dependence*

The frequency dependence of the two greases was studied at four different temperatures using a reasonably constant deformation amplitude ($\gamma = 8\%$). The results for greases A and B are shown in Figures 2 and 3, respectively. From these figures it can be seen that the moduli G_1 and G_2 increase only by a factor of about 10, while the frequency is increased ten thousandfold. In the case of grease A, it is interesting to note that the G_1 remains larger than G_2 over the whole frequency range and at temperatures below 35°C. With grease B, G_1 becomes consistently smaller than G_2 at higher frequencies and at all temperatures tested. This indicates that the viscous response of this grease increases consistently in importance and predominates at higher frequencies.

C. *Amplitude Dependence*

The amplitude dependence of the two greases was examined at various frequencies and at three temperatures, −3°, 25° and 50°C. The results are shown in Figures 4 and 5 for greases A and B, respectively. In both cases a very strong dependence on the extent of deformation is noted, which is quite different from the previous observation made of the frequency dependence. From a comparison of these two figures, one notes again that for grease B, G_1 decreases in magnitude more rapidly than G_2 indicating that the viscous response increases with increasing deformation.

From the shape of the curves and the photographic pictures obtained in determining the amplitude dependence of the two greases, it was noticed that there existed a non-linear region, i.e. a region in which the response of the materials to sinusoidal stressing was non-linear. This appears in the figures as a change in slope and in the photographs as a distortion of the normally observed ellipse. This region of non-linearity seems to extend from very low shear strains of about 0.3 per cent to about 5 per cent and occurs for both greases in approximately the same region. A harmonic analysis was made to determine whether the stress or the strain or both are non-linear. This was done by separating the stress and strain curves and analyzing each one separately. To obtain higher sensitivity for this study, grease B was tested at −3°C at 0.0034 cycles per sec. The reason for selecting grease B for the study was based on the fact that it showed the stronger non-linear response of the two samples. The low frequency of

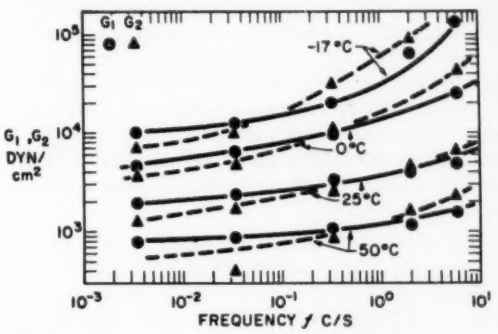


FIGURE 3, frequency dependence of grease B.

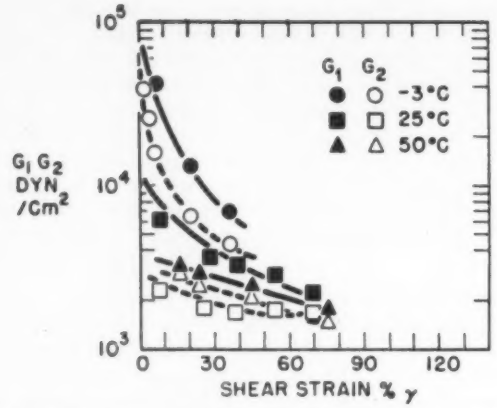


FIGURE 4, amplitude dependence of grease A (Frequency = 0.34 cps).

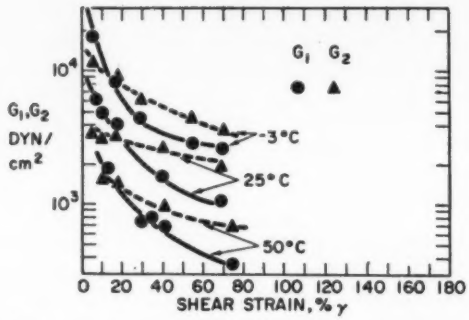


FIGURE 5, amplitude dependence of grease B (Frequency = 0.34 cps).

0.0034 cycles per sec was selected because it permitted the use of a two-pen recorder to trace force and deformation versus time. These curves are shown in Appendix 2 together with the details of the calculations. The deformation curve was found to be perfectly sinusoidal and, therefore, the entire non-linearity had to be present in the force curve. It was also established that this observed non-linearity was not due to any superposition of higher harmonic motions but was characteristic of the fundamental wave. This confirmed the assumption on which the procedure outlined in Appendix 1 is based—namely, that only fundamental waves were being measured and that the results so obtained can be used to calculate the stress as the function of strain. The surprising factor of this analysis was the indication that the non-linearity resides in the loss modulus, the viscous behavior, which is contrary to what one usually expects.

D. *Temperature Dependence*

As was noticed already in Figures 2 and 3, an increase in temperature caused a decrease in the value of the storage and loss moduli over the whole frequency range indicating a decrease in rigidity and viscous response. A similar condition prevailed when the amplitude dependence of the grease was tested at different temperatures as was shown in Figures 4 and 5. An additional peculiarity that could be observed was the fact that for grease A an increase in temperature to 50°C increased rather than decreased the value of the loss modulus over that at 25°C, particularly at lower shear strains. The amplitude dependence of the two greases remained quite similar at different frequencies as shown in Figures 6 and 7 for greases A and B, respectively. From these figures it can be seen that increasing the frequency over the same range of shear strains produces essentially similar effects to decreasing the temperature.

The strong amplitude dependence evidenced by these two greases make it somewhat difficult to compare their true frequency dependence. Ideally, one would have to examine the greases at all frequencies at 0 amplitude. Practically, this is not possible but one could use very small amplitudes and assume them to be practically negligible. Unfortunately, when working at these low frequencies, it is impossible to achieve this condition because the greases are too soft and the instrument is not sensitive enough to resolve the small forces necessary. Also, one is more interested in the behavior of the greases at large deformations and hence large amplitudes, since these represent more realistic conditions under which the greases are expected to operate.

E. *Effect of Geometry*

Two different clearances were used in performing the

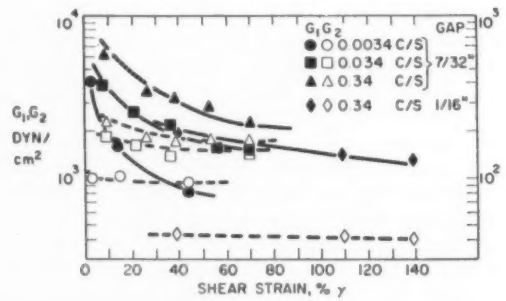


FIGURE 6, effect of frequency and geometry on amplitude dependence of grease A (25°C).

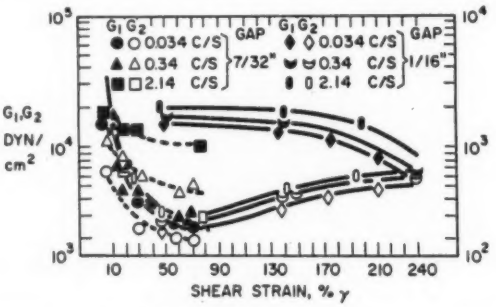


FIGURE 7, effect of frequency and geometry on amplitude dependence of grease B (—3°C).

series of experiments. In one case a wide opening of 7/32 of an inch was used, while in the other case a relatively narrow gap of 1/16 of an inch was used. The resulting differences in amplitude dependence have been incorporated into Figures 6 and 7. It can be readily seen that by narrowing the gap the greases will be much more severely deformed and hence more structural breakdown occurs. Under these conditions, the greases have smaller storage and loss moduli than when the wide opening is used, indicating considerable softening. With this narrow clearance, one cannot approach properly the region of small deformations necessary for the investigation of the region on non-linearity. But these narrow clearances can be used to point out the behavior of the test greases under large deformations and indicate unusual occurrences in that region. This point is clearly shown in Figure 7 for grease B. It can be noticed that G_2 remains smaller than G_1 over a wide range of deformations and increases only at very large deformations, whereas G_2 for grease A remains essentially unchanged. It is for this reason that these values of G_1 and G_2 have not been shown over the same extended range for grease A. Thus, results obtained with these two clearances supplement each other and provide information over a wide range of deformations.

F. *Principle of Reduced Variables*

In the preceding discussion, it was pointed out that increasing the frequency or decreasing the temperature produced similar results. This trend suggests an equivalence between frequency and temperature. Ferry[9] recognized this relationship and introduced the concept of "reduced variables" in which he assumed that each mechanical response of the system has an identical temperature coefficient. The validity of reduced variables has been experimentally verified for many systems, particularly polymers, and permits measurements at different temperatures and different frequencies to be reduced to one temperature, in this case 25°C, while covering a much wider range of frequencies.

In practice the isothermal curves of G_1 and G_2 are plotted against the frequency on a log-log plot. The "reduced" curve is produced by sliding each isothermal curve along the frequency axis until the points line up with the reference curve at 25°C to give a continuously smooth curve. The amount of horizontal shift necessary to obtain coincidence is a measure of the temperature dependence of the material and is referred to as the shift factor, log a_T.

An attempt was made to apply this principle to both greases. While it was possible to do this reduced variable treatment for grease A, it was not applicable for grease B under prevailing circumstance. Apparently mechanical responses, the reactions of the material to external deformation are not equally affected by temperature changes. One cannot shift these curves parallel to each other as can be readily appreciated from Figure 3. The graph of the reduced variable plot for grease A

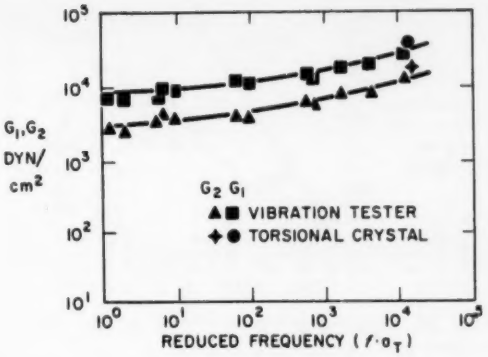


FIGURE 8, reduced curve, 25°C grease A.

is shown in Figure 8. One can see that G_1 is always larger than G_2 over the extended frequency range.

In order to check the validity of the method of reduced variables for this grease system, the dynamic behavior of grease A was measured at high frequencies with a torsion crystal using the Mason technique.[10] The torsion crystal was brought into contact with the grease with the bottom of the crystal just touching the material to be measured. The technique and the evaluation are outlined in the literature.[11] The ambient temperature of the measurements was 28.5°C and the shear was so small that it was assumed to be 0. In order to compare these results with the ones obtained in the vibration tester, it was necessary to correct for the shear dependence using the results obtained previously from the amplitude dependence. After this correction and the correction for different temperatures were made, it was found that the moduli so found fit very closely on the reduced frequency curve as shown in Figure 8.

An interesting piece of information from this reduced variable principle can be obtained by plotting the shift factor log a_T versus $1/T$. The slope of this curve yields an apparent activation energy of viscous flow. The physical meaning of this activiation energy is somewhat vague at the present time since it refers to

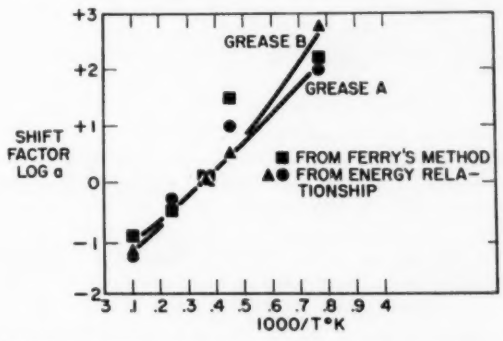


FIGURE 9, temperature dependence of test greases.

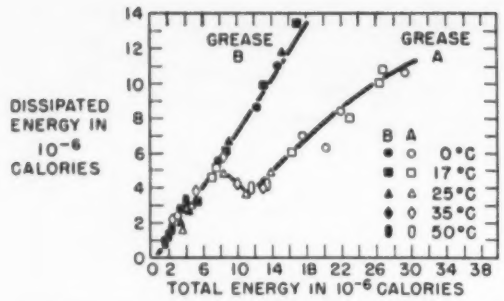


FIGURE 10, relationship between total energy and dissipated energy.

moles of flow units, and it is impossible to define the meaning of moles in this case. This plot is shown in Figure 9, indicating that there exists an essentially straight line relationship between 0 and 50°C. The slope of this line indicates an activiation energy of about 22 kcal/mol.

G. *Relationship of Total to Dissipated Energy*

Another interesting piece of information can be obtained from a study of the relationship that exists between the total energy expended and energy dissipated per cycle. These data have been obtained using the Equations [7] and [8], given in part 2, and are shown in detail in Figure 10 for both greases. For grease A one can see that there exists a maximum at low energy input. At higher energy inputs the dissipated energy increase is practically linear. In the case of grease B there is a continuous increase in the relationship between energy input and energy dissipated indicating that a different mechanism is involved in the mechanical response of this system to strain. This is another way of confirming the previous statement that the two greases respond differently to deformation forces. In both cases the curve appears to start somewhat to the right of the common zero point, indicating that no flow will occur until a finite amount of energy is put into the system. From an extrapolation of both curves to the abscissa one gets approximately 30 dyn/cm² as the minimum stress necessary to produce flow.

This method of plotting energy input against energy dissipation can also be used to verify the concept of reduced variables previously advanced. From the amount of overlap that exists between measurements made at the same frequencies but at different temperatures, a similar shift factor can be deduced which when plotted against $1/T$ gives identical results with the one obtained previously for grease A as shown in Figure 9. Such procedure yields also information on the thermal behavior of grease B where the method proposed by Ferry does not work because of the peculiar shape of the frequency isotherms, as shown also in Figure 9. From this plot, the activation energy of grease B is found to be roughly the same as that for grease A—namely, 22 kcal/mol between 17 and 50°C. Below that temperature the relationship between shift factor and reciprocal temperature are no longer linear and the slope increases rapidly to much higher values. This is considered further evidence of the aforementioned different behavior of grease B as compared to grease A.

4. Interpretation and Discussion of Experimental Results

Once the response of the two greases to time dependent stresses had been experimentally determined, it became desirable to establish the extent to which these results reflect the known properties and performance characteristics. Also, it was felt that these results should be examined to see whether they could be used to classify the behavior of greases in terms of other well-defined systems such as polymers.

A. *Comparisons of Vibration Tester Results with Known Physical Properties*

The vibration tests were performed at low shear rates, consequently only grease properties, such as consistency, which are measured at low shear rates would be expected to correlate.

1. *Consistency*—The effect of small deformations in the vibration tester can be compared to the effects produced in the penetration test as shown in Table 4.

TABLE 4
Comparison of Vibration Test Results with Penetration Results

Properties	*Grease A*	*Grease B*
Unworked penetration at 25°C, in mm/10	275	274
Extrapolated value of G_1 at 0% γ at 25°C, 0.34 c/s, in dyn/cm²	1.1×10^4	1.1×10^4
Minimum stress in dyn/cm² to produce movement (τ_1)	30	30
Change in penetration after 60 strokes in ASTM grease worker at 25°C mm/10	5	36
$d(\log G_1)/d\gamma$ for small values of γ at 25°C	−0.16	−0.23

The similar consistency of the two greases as evidenced by their unworked penetration is reflected in two values which were obtained from the amplitude dependence of the greases in one case and the energy relationship (see Fig. 10) in the other case. The change in penetration upon working is well reflected in the rate of change of log G_1 with deformation which is determined from the amplitude dependence of these two greases. If the measurements were carried out to bigger deformations, they would probably permit further correlation with prolonged working in the ASTM grease worker.

2. *Temperature Effects*—The effect of temperature on the behavior of the two greases as determined in the vibration tester compares quite favorably with

Continued on page 227

Previews of the 27th NLGI Annual Meeting

Roosevelt Hotel
New Orleans, La.
Oct. 25-26-27-28, 1959

You Are Invited–

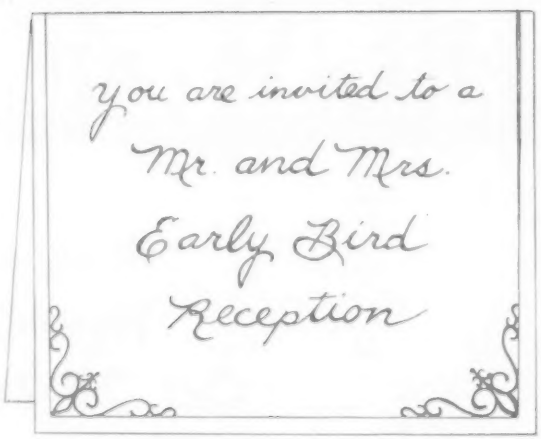
27th Annual Meeting
Oct. 25-28, 1959

NLGI invites you to come to New Orleans! The 27th Annual Meeting of the National Lubricating Grease Institute will be an exciting combination of places to go, things to do, and the largest and most extensive program in the history of the Institute. Special emphasis has been given to current industry interests (see page four for a listing) and the subjects covered will be presented by experts in their fields.

General sessions will spotlight the chassis lubricant market, and project into the future on a number of subjects. A symposium on manufacturing procedures and equipment has been designed for maximum interest, with a second symposium on dispensing problems created to deal with this important phase. Questions and answers, informal discussions, sub-committee sessions . . . more than 500 industry experts representing the industry from an international standpoint will meet October 25, 26, 27, 28 at the Roosevelt Hotel. For the latest in testing, scientific developments and marketing, we hope you will be a part of NLGI in New Orleans, 1959.

It's a New Function . . . Come Meet Your Friends at the "Early Bird Party"

SOMETHING new has been added to the Annual Meeting! Through the courtesy of NLGI Associate members, a complimentary "Mr. and Mrs. Early Bird Reception" will be held Sunday evening, October 25, at the Roosevelt Hotel. Members may get acquainted with new friends and old acquaintances during the reception, 6 to 7:30 p.m., and still have time to visit parts of historic New Orleans. Member firms participating include . . . American Can, American Flange, Balcrank, Bennett Industries, Baker Castor Oil, Godfrey L. Cabot, Central Can, Continental Can, Darling, Foote Mineral, Harchem, Humko, Jones & Laughlin, Lubrizol, Rheem Manufacturing, Stratford Engineering, United States Steel, Witco Chemical and Sumner Sollitt. From there it's just a short step to the "Paris of America"!



Progress Reported, New Areas Charted

THE year of intensive planning by committees concerned with NLGI in New Orleans has been filled with an intense effort to offer a program which fits the needs of the Institute as expressed by the members. And there are other benefits . . . the meetings.

FUNDAMENTAL approaches to problems through general sessions and committee work are always the main business of an Annual Meeting. The regular Technical Committee on Wednesday Morning, October 28, will see a giant growth step for the Institute realized when the Sub-committees, new, reorganized, and the traditional groups, make their reports and recommendations. With scope of the Technical committee matching the enlarged interests of the Institute and growing industry technological development, the sessions in New Orleans will mean the focusing of much research data, methods, tests, etc., and allow the membership to prepare for greater demands which are resulting from continual new developments.

MARKETERS will hear papers on new product ideas, the international lubricant situation, container expectations in the future, and current status of the non-metallic, non-lubricated bearing. Members and friends of NLGI are both invited to avoid last minute disappointment by placing reservations at the Roosevelt Hotel in New Orleans now, and insure adequate accommodations.

The Ladies Are Most Welcome— Special Women's Morning Program Planned

SCENIC New Orleans means a different type of Annual meeting, with many wives attending. The ladies are most welcome . . . a special program has been arranged during the general sessions, which will give them time for a good look at this famous city, yet allow for individual activities during the day. After the "Mr. and Mrs. Early Bird Reception" Sunday evening, wives can spend mornings beginning with get-acquainted coffees and tours of historic New Orleans. Picturesque old market places, shops, plantations, homes . . . with luncheons at famous restaurants, and plenty of time for shopping afterward! NLGI evening social hours and banquets are scheduled to conclude in time for visits to the gay and colorful night life for which the city is famous. Bring the ladies to New Orleans!

Schedule of NLGI Annual Meeting Papers

General Session

KEYNOTE ADDRESS, *Dr. Ralph Robey*, Economic Advisor to the National Association of Manufacturers, New York

TEN YEARS IN RETROSPECT, *T. G. Roehner*, Socony Mobil Oil, New York

CURRENT STATUS OF THE NON-LUBRICATED, NON-METALLIC BEARING IN THE AUTOMOTIVE INDUSTRY, *J. W. Lane*, Socony Mobil Oil, New York

ROAD EVALUATION OF AUTOMOTIVE CHASSIS GREASE, *E. W. Tracy, Jr.*, Southwest Research Institute, San Antonio, Texas. Co-Author: *E. E. Smith*, Climax Molybdenum, New York

WEAR RATES IN CHASSIS LUBRICATION OF PASSENGER CARS, *W. L. Hayne, L. C. Brunstrum*, Standard Oil (Indiana) Whiting, Indiana

MORE SALES—FOR YOU AND YOUR INDUSTRY, *J. Y. McCollister*, McCollister Grease & Oil, Omaha, Nebraska

REVIEW OF THE LUBRICATING GREASE INDUSTRY IN EUROPE AND LATIN AMERICA, *D. C. Sandy*, Standard Oil (New Jersey), New York

A MODIFIED CLAY THICKENER FOR LUBRICATING FLUIDS, *R. E. Fariss*, Baroid Division, National Lead, Houston

A STUDY OF OPEN GEAR LUBRICATION, *P. O. Malloy, Cloyce Daniels*, Esso Research, New York

GREASE CONTAINERS—PAST, PRESENT AND FUTURE, *W. R. Hosler*, Battenfeld Grease and Oil, Kansas City

RETENTION OF LIQUIDS IN SOAP/HYDROCARBON SYSTEMS, *A. J. Grozek, G. H. Bell*, Petroleum Division, British Petroleum Research Centre, Middlesex, England (To be presented by K. S. Cuddington, BP North America)

WEAR RATE IN LUBRICATION OF BUS CHASSIS, *J. Margetic*, Chicago Transit Authority, Chicago

Manufacturing Symposium

GREASE MIXER DESIGN, *K. G. Timm*, Struthers Wells, Warren, Pennsylvania

A PHILOSOPHY OF GREASE MILLING, *Melville Ehrlich, F. S. Sayles*, American Lubricants, Buffalo, New York

MATERIAL HANDLING AND PROCESSING AIDS IN THE MANUFACTURING OF GREASE, *S. E. Hendricks, R. S. Stoops*, International Lubricants, New Orleans

A NEW TECHNIQUE FOR CONTINUOUS MEASUREMENT OF GREASE CONSISTENCY DURING MANUFACTURE, *J. L. Dreher, C. F. Carter*, California Research, Richmond, California

Dispensing Symposium

DEVELOPMENT OF A CHART FOR PREDICTING THE PUMPABILITY OF GREASES, *W. L. Gabbert*, Lincoln Engineering, St. Louis

WESTINGHOUSE GREASE DISPENSING PRACTICE, *R. C. Garretson*, Westinghouse Electric, East Pittsburgh

REVIEW OF RESEARCH STUDIES SHOWING NEED FOR A VISCOSITY TEST BELOW 1 SEC. -1, *E. L. Armstrong*, Socony Mobil Oil, Brooklyn, New York

A REVIEW OF NLGI'S TENTATIVE DISPENSING METHOD, *R. D. Skoglund*, Texaco, Beacon, New York

BULK GREASE HANDLING, *C. A. Bailey*, National Tube Division, United States Steel, Pittsburgh

OTHER ANNUAL MEETING HIGHLIGHTS include Sub-Committee meetings, Steering Committee action, the Annual Business Meeting, and the NLGI Technical Committee meeting.

NLGI 27TH ANNUAL MEETING, ROOSEVELT HOTEL, NEW ORLEANS, OCTOBER 25, 26, 27, 28, 1959. REGISTRATION FIVE DOLLARS (MEMBERS) AND TEN DOLLARS (NON-MEMBERS), BANQUET TEN DOLLARS AND SOCIAL HOUR THREE DOLLARS. FOR FURTHER INFORMATION PLEASE CONTACT: NATIONAL LUBRICATING GREASE INSTITUTE, 4638 J. C. NICHOLS PARKWAY, KANSAS CITY 12, MISSOURI. (PHONE) VALENTINE 1-6771. T. W. H. MILLER, GENERAL MANAGER.

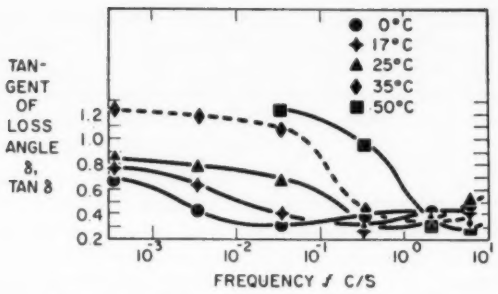


FIGURE 11, effect of temperature on loss angle—frequency relationship, grease A.

the change in unworked penetration and apparent viscosity with temperatures as shown in Table 5.

TABLE 5
Comparison of Observed Temperature Effects

	Grease A	*Grease B*
Increase in unworked penetration, in mm/10 (increase in temperature from 25 to 100°C)	75	26
Change in apparent viscosity with temperature (between 25 and 100°C)	large	small
Shift in loss angle minimum, in cycles/sec (increase in temperature from 0° to 50°C) (See Figs. 11 and 12)	0.024 to 4.4	0

It should be recalled that the tangent of the loss angle represents the ratio of loss modulus to storage modulus and hence represents the ratio of energy dissipated to energy stored per cycle. Since the loss angle-frequency relationship does not shift with temperature for grease B, the structural changes responsible for the movement of this grease appears to be unaffected by temperature changes. This suggests that the results from the vibration tester might be used to uncover in more detail the relationship that exists between the structure of greases and their physical properties.

3. *New Information*—The data presented earlier were analyzed with regard to possible new information that could be obtained from them and several interesting observations were made.

(a) *The existence of two yield stresses*—It has been shown earlier in discussing the relationship between total energy input and amount of energy dissipated, that both greases require a minimum stress τ_1 of 30 dyn/cm² to produce movement. On the other hand, an analysis of the region of non-linearity of the stress-strain relationship indicates the existence of a "yield stress" of 560-650 dyn/cm² for grease A and 670-710 dyn/cm² for grease B. These data indicate the existence of two yield stresses, τ_1 and τ_2, and hence two types of grease movement, namely bulk movement and

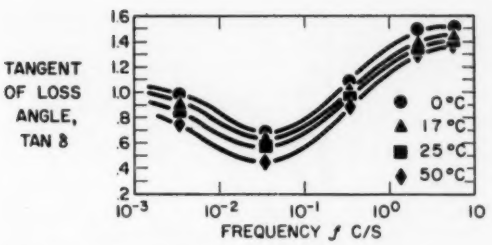


FIGURE 12, effect of temperature on loss angle—frequency relationship, grease B.

plastic flow. If one keeps in mind the fact that any movement of grease will require a rupture of contact points among soap fibers, one can explain these two types of movement as follows: Between τ_1 and τ_2 the rate with which contact points are broken is smaller than the rate with which these contacts reform or heal, so that the net effect is the movement of the grease in bulk or creep. At τ_2 the two rates become equal, and above τ_2 the rate of rupture exceeds that of healing. As the shearing stresses further increase, these ruptures lead to smaller and smaller soap fiber aggregates until the apparent viscosity of the grease will approach that of the base oil. The apparent similarity of the behavior of the two greases at the small deformations produced in the vibration tester is not surprising since these deformations are not expected to produce any noticeable damage to the soap structure. One would expect that the structural differences become more noticeable at higher stresses or at lower stresses applied at elevated temperatures.

(b) *Relaxation time distribution*—From Figure 10, it can be seen that about 40 to 90 per cent of the total energy was dissipated under the prevailing experimental conditions. This means that from 60 to 10 per cent of the total energy has been stored in a more or less reversible fashion. This energy can be stored by stretching bonds, changing bond angles, coiling or uncoiling molecular aggregates or just simply displacing molecules or molecular aggregates from their equilibrium position. After the deforming stresses have been released, these displacements and molecular changes will be reversed within a definite time interval. This time interval is called the relaxation time and the molecular changes involved are referred to as relaxation mechanisms. In complex systems such as greases, there are many ways and means of storing energy and hence a large number of relaxation times. From the results obtained with the vibration tester, one obtains an approximation of the distribution of relaxation times, $[H(t_r)]$, using Ferry's first approximation method'. The results are shown in Figures 13 and 14 for greases A and B, respectively. It is immediately apparent that the two greases behave differently. Grease A has a rather sharp well-defined maximum similar to those observed in amorphous polymers which shifts

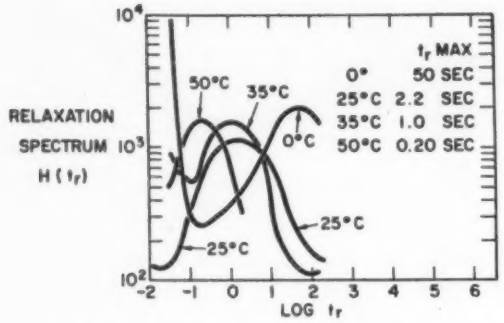


FIGURE 13, relaxation spectrum of grease A.

with temperature from 50 sec at 0°C to 0.2 sec at 50°C, while grease B has a maximum at 1.5 sec that is barely visible at 17°C, is well developed at 25°C and again hardly noticeable at 50°C and does not shift with temperature. Such behavior is believed to be unique among polymers as well as greases and indicates that temperature changes have only a very small effect on this material.

The shape of the relaxation distribution curves suggests the existence of two further peaks both at longer and shorter times. Relaxation times of 1600 and 10,000 sec have been previously reported (4) for sodium and lithium soap greases, respectively, when exposed to small compressive deformations. Eyring[12] reported relaxation times of 3 sec at 30°C for an AN-G-25 type grease. These data were obtained in concentric cylinder type viscometers. No data have as yet been reported on the very shortest relaxation times.

The significance of the observed relaxation time in terms of molecular processes can only be guessed at as yet. It is possible that this time interval represents the movement of soap aggregates from one equilibrium position to an adjacent one. In the two greases these soap aggregates are quite different in size and accordingly will take different times to carry out this movement. This tentative assignment of this movement of soap aggregates is made largely on the basis of the

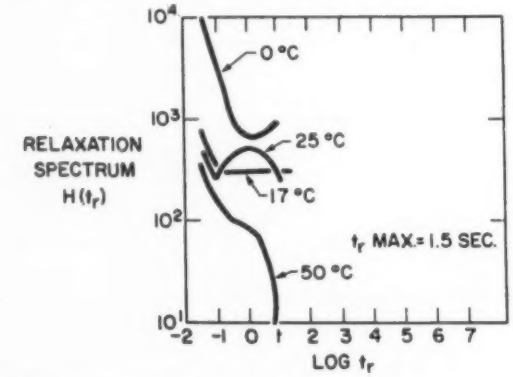


FIGURE 14, relaxation spectrum of grease B.

electron microscope evidence available. As has been shown in Figure 1, grease A has the long thin fibers typical of regular soap type greases while grease B has short, well-formed needle-like fibers that do not wrap around each other. The displacement of such needle-like fibres will be quite different from those long fibers which behave like an amorphous polymer.

B. *Comparison of Greases with Polymers*

Once the response of these two greases to time dependent stresses had been experimentally determined, it became desirable to classify this behavior in terms of other well-defined systems such as high polymers. A comparison of greases and high polymers had been suggested earlier when studying the structure of greases.[5] If one makes, therefore, this comparison one finds that greases resemble amorphous linear polymers, which also show a relatively small frequency and large amplitude dependence. Like greases, these polymers show a large amount of non-linear, irreversible behavior. An examination of the structural differences existing between greases and amorphous linear polymers suggests the possible explanation of their similar mechanical behavior.

Obviously, greases resemble linear polymers in as much as most soaps usually form long thin fibers. Greases also resemble amorphous polymers because these fibers do not always line up in a geometric fashion but are randomly oriented and entangle with each other. While the crystalline regions within the fiber remain intact, the space occupied by the fibers is not crystalline. The forces holding this network of fibers together will be similar to those holding polymer molecules together except for the additional contribution of the polar groups in the soap fiber which will provide for additional attractive forces in the case of greases. One might, therefore, expect some advantage in this respect over the amorphous polymer. On the other hand, the polymer molecules themselves are made up of monomers which are held together by strong chemical bonds. In the soap fibers, only ionic type forces together with van der Waals forces help to hold the soap molecules together. This condition would give a greater structural stability in at least one direction to the polymer system, which indeed they are known to have. The net effect of the situation is difficult to assess, but one would expect that these differences just pointed out would not cause significant differences in the mechanical properties of these two systems. The effect of fiber length can be held responsible for the different behavior of the two test greases. The short fibers of grease B cannot entangle like those of grease A and any movement will depend more on the forces holding the fibers together than on their disentanglement.

5. Summary and Conclusions

On the basis of the experimental results, it can be concluded that the vibration tester method, developed for the study of viscoelastic materials, can be successfully applied to greases. It was shown that the moduli

of greases change little with frequency but change considerably with changes in amplitude and temperature. Decrease in the clearance used in deforming the grease were found to decrease the value of the moduli and reflect the shear sensitivity of greases.

Since the vibration tester operates at low shear rates, only those known grease properties that are measured at similar low shear could be correlated with the experimental results. Thus, the consistency of the two greases as reflected in their unworked penetration was correlated with the extrapolated value of the modulus of rigidity, G_1, and with the minimum stress required to produce grease movement. Similarly the effect of working the greases for 60 strokes in the ASTM worker was correlated with the rate of change of log G_1 with increasing deformation. The effect of temperature on the consistency of the two greases was found to be reflected in the shift of the loss angle tangent with temperature.

In addition to these correlations, it was established that both greases have two yield stresses which reflect the existence of two types of movement, bulk movement and plastic flow. In the bulk movement, fiber contacts are broken, but reform at such a fast rate that the bulk of the grease remains unchanged. In plastic flow, contacts are broken faster than they can reform and the bulk of the grease will soften.

The structural differences existing between the two greases are particularly reflected in the change of the relaxation time distribution with temperature. While grease A has a maximum that shifts with temperature from 50 sec at 0°C to 0.2 sec at 50°C, grease B shows only a clear maximum of 1.5 sec at 25°C which does not shift with temperature but merely tends to disappear with both increasing and decreasing temperature.

The behavior of the two greases in the vibration tester resembles that of amorphous linear polymers. This resemblance is particularly striking for grease A, while it is less pronounced for grease B. This is taken as further confirmation of the difference in soap fiber structure that exists between the two greases. Thus the vibration tester confirms the significance of the relationship between structure and physical properties of greases. More detailed work is now being planned to establish this relationship more quantitatively.

6. Acknowledgment

The authors wish to acknowledge the help of Dr. W. Philippoff of the Franklin Institute in performing the experimental part of this investigation and the many suggestions that have been advanced by him and his co-workers in support of this project.

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Appendix 1

Calculation of instrument constant and moduli

The formulas used for the calculation of the maximum shearing stress τ_0 and the maximum shear strain γ_0 are for this arrangement:[13, 14]

$$\tau_0 = \frac{F}{2\pi L R_i} \cdot \frac{1-c^2}{1+c^2}$$

$$\gamma_0 = \frac{D}{R_i} \cdot \frac{1-c^2}{1+c^2} \cdot \frac{1}{\ln \frac{1}{c} - \left(\frac{1-c^2}{1+c^2}\right)}$$

where R_i = radius of inner cylinder (plunger in cm).
$c = R_i/R_0$ and R_0 is radius of outer cylinder (cup), in cm.
D = deformation, displacement of inner cylinder with respect to outer cylinder, in microns.
F = force required to move inner cylinder with respect to outer cylinder against the drag

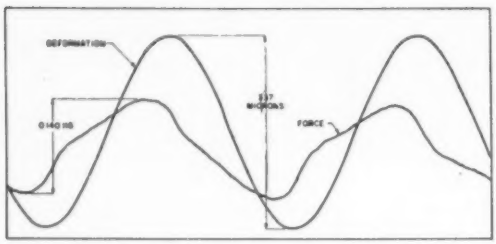

FIGURE 15, force—deformation relationship for grease B (0.0034 cps, —3°C).

of the material contained in the gap R_0-R_i, in kg.

L = length of inner cylinder in cm.

since $|G| = \tau_0/\gamma_0$ it follows that $|G| = E/D \cdot K$ where K is the instrument constant and given by the expression

$$K = \frac{1}{2\pi L} \cdot \left[\ln \frac{1}{c} - \left(\frac{1-c^2}{1+c^2} \right) \right]$$

and has the dimensions of (dyn/cm²) × (microns/kg).

The values of F and D are obtained from the geometry of the hysteresis loop as shown in Figure 16.

Appendix 2

Harmonic analysis of non-linear response

Using a two-pen recorder, traces of force and deformation as a function of time were obtained for grease B at 0.00340 cycles per sec and −3°C. These curves are shown in Figure 15 and indicate that the deformation curve is perfectly sinusoidal. The entire non-linearity is present in the force curve. A harmonic analysis of this curve gave the following relationship:

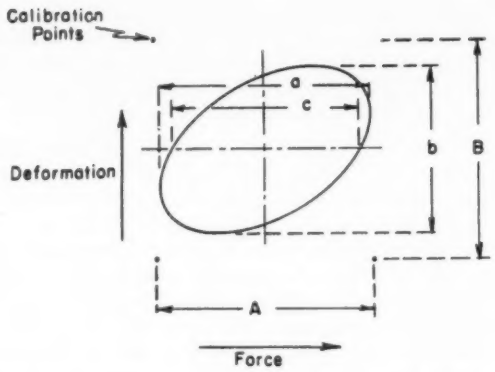

FIGURE 16, stress—strain ellipse. (Thus F = a/A, D = b/B, sin δ = c/a, and G_1 = (KF cos δ)/D, G_2 = (KF sin δ)/D).

$$F = 61{\cdot}0 \sin(\omega t + 52°40') - 7{\cdot}6 \sin(3\omega t + 83°15') + 1{\cdot}04 \sin(5\omega t + 80°38'')$$

or approximately

$$F = 37 \sin \omega t + 48{\cdot}5 \cos \omega t - 7{\cdot}6 \cos 3\omega t + 1{\cdot}04 \cos 5\omega t$$

where the constants are in millimeter of travel on the graph, ω is the angular frequency, and t the time in seconds.

If the usual approximation is used, of taking the peak values and one measures the phase shift of these peaks, one obtains

$$F = 62 \sin(\omega t + 53{\cdot}0°)$$

in good agreement with the result of the harmonic analysis. The presence of the higher harmonics as cosine terms indicates that the non-linearity resides in the loss modulus rather than the storage modulus. ■

About the Authors

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J. J. Kolfenbach *obtained the BA degree from Loras college in 1939 and his PhD in biophysical chemistry from Iowa State college in 1941. He has been associated with the Esso Research and Engineering company since 1944. He is presently section head of the industrial lubricants section. He is a member of ACS, Sigma Xi, and SAE. A long - time contributor to the NLGI SPOKESMAN, he is also a former chairman of the Technical Subcommittee on Fundamental Research and is now Assistant Head of the Special Projects Unit.*

Type A, and Type A, Suffix A, Automatic Transmission Fluids

By: W. H. Kite, Jr.
Texaco, Inc.

Presented at the API Lubrication Committee Meeting in Detroit, February, 1959

ALTHOUGH IT WAS not realized at the time, the introduction of the Oldsmobile automatic safety transmission in 1937 established the need for an entirely new type of lubricant. At first, an attempt was made to lubricate this transmission with motor oils which were of the straight-mineral type, with the exception that some contained oxidation inhibitors. This choice was logical because the engines of that time were lubricated satisfactorily with such oils and, moreover, little was known concerning additive-type fluids. However, service experience soon showed that a close relationship existed between automatic transmission fluid quality and actual transmission performance. Use of the straight-mineral type oils often resulted in sludge and varnish formation. Oil deterioration frequently progressed to the extent that transmission performance became sluggish, and, in some cases, the units became inoperative.

The introduction of the Hydra-Matic transmission in 1939 served to emphasize the deficiencies of the straight-mineral type oils and other fluids of the day. As the shortcomings became more obvious and as it became apparent that the number of automatic transmissions in use would be increasing yearly, General Motors research began work on the necessary steps preliminary to the formulation of a specification which, for the first time, would define the specific characteristics and properties desired of automatic transmission fluids and which would list the tests necessary to insure that the fluids would fulfill these requirements.

The necessity for a definitive specification covering automatic transmission fluids derives from the very complex nature of this type fluid. In fact, the automatic transmission fluid is generally considered as probably one of the most complex lubricants in existence. This viewpoint can be justified through consideration of the many requirements placed upon the fluid by the intricate mechanisms which comprise the modern automatic transmission.

The complexity of the automatic transmission fluid is due, in a large part, to the fact that it must serve a number of functions in the automatic transmission. These functions can be summarized generally as follows:

1. Serve as a medium for transmitting power in the fluid coupling.
2. Serve as an hydraulic medium for an extremely complex hydraulic control system.
3. Serve as a medium for transferring and dissipating heat.
4. Serve as a lubricant for a variety of mechanisms, such as bearings, gears, clutches, etc.

In order to perform the above functions successfully, the fluid obviously must possess a number of special characteristics. In 1949, General Motors Research issued the first Type A specification, which defined

the necessary characteristics and which listed the test procedures to be employed in insuring the fluids possessed these characteristics.

In the program for qualifying fluids against the Type A specification, General Motors Research enlisted the services of the Armour Research Foundation of the Illinois Institute of Technology. Arrangements were made for some of the qualification tests to be conducted by Armour and some by General Motors. Fluids so qualifying were identified by an "Armour Qualification Number" (AQ-ATF——), the blank after the ATF being filled in with the specific qualification number, commonly called "AQ Number," assigned to the fluid. This identification, labeled on each container, assured the customer that unsatisfactory fluids had been eliminated and, at the same time, provided him with the means of identifying qualified fluids.

In 1951, the original Type A specification was modified, particularly with respect to viscosity, viscosity index, flash point and fire point, to make it more realistic in the light of experience gained during the intervening two years. This 1951 version of the specification remained essentially unchanged until November 1, 1957.

The 1951 Type A specification required that the "Research Technique for the Study of the Oxidation Characteristics of Crankcase Oils" (CRS Designation L-4), more popularly known as the Chevrolet L-4 test, be used as the method for evaluating the high temperature oxidation resistance of Type A automatic transmission fluids. Two L-4 tests were conducted, one on the undiluted fluid and one on the fluid diluted with an equal portion of standard straight mineral oil.

Although the Chevrolet L-4 test proved to be generally satisfactory, there were several shortcomings apparent in its application for establishing the oxidation resistance of automatic transmission fluids:

1. Testing in an engine subjects the fluid to contamination by fuel combustion products which are not encountered under service conditions in a transmission.
2. Use of the arbitrarily specified mineral oil in the dilute L-4 phase constitutes an artificial requirement and thereby may result in an inaccurate evaluation of fluids containing additives not compatible with the reference oil.
3. The operating conditions and requirements, indeed, the very mechanical construction of any engine are greatly dissimilar to those of an automatic transmission.
4. The Chevrolet L-4 test operating conditions are not sufficiently severe to comprise a basis for predicting accurately the high-temperature oxidation performance of automatic transmission fluids when subjected to service in modern transmissions.

With the increase in automatic transmission complexity and engine horsepower, the lack of severity of the Chevrolet L-4 test became increasingly apparent. Beginning with some 1956 models, instances were encountered in which the use of automatic transmission fluids fully qualified under the existing Type A specification resulted in sludge formation under severe service conditions in the new transmissions. As a result, General Motors issued a new specification dated November 1, 1957. This new specification, shown as Table I, is identified as "Automatic Transmission Fluid, Type A, Suffix A, Identification Specification." The methods of test are outlined in Table IA.

TABLE I

Automatic Transmission Fluid, Type A
Suffix A, Identification
Specification Requirements

November 1, 1957

a. Miscibility	Pass
b. Viscosity, Saybolt Universal, 210°	49 seconds, min.
c. Flash Point, °F.	320 minimum
d. Fire Point, °F.	350 minimum
e. Low Temperature Fluidity	Pass
f. Copper-Strip Test, 3 hrs. at 300°F.	Pass
g. Anti-Foaming Properties	Pass
h. Heating Test	Pass
i. Non-Corrosion and Non-Rusting Properties	Pass
j. Effect on Seals	Pass
k. Odor	Pass
l. Non-Toxic Properties	Pass
m. Oxidation Test on Fluid as Marketed	Pass
n. Non-Chatter or Squawking Properties	Pass
o. Durability (Cycling) Test	Pass
p. Viscosity Stability, viscosity during and at conclusion of durability (Cycling) and performance tests, Saybolt Universal, 210°F.	46.5 seconds, min.
q. Frictional Properties	Pass
r. Performance in Transmissions Under Service Conditions	Pass
s. Performance in Transmission Installed in Car and Tested in Cold Room at −10°F.	Pass

This latest specification differs from the preceding Type A specification primarily from the standpoint that the "Research Technique for Determining the Oxidation Resistance and Thermal Stability of Power Transmission Fluids" (CRS Designation L-39) has

TABLE IA

Methods of Test

The following methods of test shall be employed in evaluating Automatic Transmission Fluid, Type A, Suffix A, Identification:

a. Determine miscibility by Armour Research Foundation Method for MIL-L-2104A crankcase oils.

b. Determine kinematic viscosity at 100 and 210°F. by ASTM Designation: D 445. Convert to Saybolt Universal by ASTM Designation: D 446.

c. Extrapolate viscosity at 0°F. by ASTM Designation: D 341.

d. Determine flash point by ASTM Designation: D 92.

e. Determine fire point by ASTM Designation: D 92.

f. Determine actual viscosity at 0, −10, −20, −30, and −40°F. with Brookfield Model LVT Viscometer with No. 4 spindle.

g. Determine copper strip corrosion by ASTM Designation: D 150. Use procedure outlined in Section 6 (a) except that sample shall be heated for 3 hours at 300°F. Report corrosion of copper strip immersed in fluid and on portion of copper strip above fluid level in terms of ASTM Copper Strip Corrosion Standards.

h. Determine maximum height of foam and time in seconds for collapse of foam in Detroit Transmission Division foam tester. (This foam tester is available to compounders of fluids at Armour Research Foundation for preliminary investigation of foaming characteristics.)

i. Determine effect of prolonged heating on evaporation loss, additive precipitation, and sludge formation.

j. Determine corrosion and rusting by ASTM Designation: D 665.

k. Determine the effect on tensile strength, elongation, volume, and cracking tendency of Acadia 3612 rubber compound by heating rubber specimens submerged in the fluid.

l. Odors that can be detected in tightly closed driver or passenger compartment of passenger cars, coaches, and/or trucks are considered objectionable.

m. Tests will be determined by nature of toxic component.

n. CLR L-39 oxidation or equivalent on fluid as marketed.

p. Determine tendency of fluid to squawk under part-throttle and under full-throttle conditions in performance test (item t.).

q. Determine durability of fluid and effect of fluid on frictional surfaces in 1956 R Oldsmobile Hydra-Matic transmission driven by 1956 Oldsmobile Super 88 or 98 engine.

r. Determine kinematic viscosity of fluid at intervals during tests (items q. and t.) by ASTM Designation: D 445. Convert to Saybolt Universal by ASTM Designation: D 446.

s. Determine smoothness of operation of fluid in transmission in performance test (item t.) and, if necessary, in OM-2 bearing testing machine to observe differences in frictional characteristics which may account for departure in performance from standard reference fluids.

t. Operate for 2,000 miles in a new Hydra-Matic transmission installed in an Oldsmobile on Proving Ground Automatic Transmission Performance Test Schedule. Observe operating charcteristics of fluid at start and again at the conclusion of the 2,000-mile schedule.

u. Operate fluid in a 1955 Oldsmobile Hydra-Matic transmission, installed in a car, in a cold room under the required temperature cycle to determine whether burning of the frictional surfaces of the composition clutch plates will occur in service in sub-zero ambient temperatures.

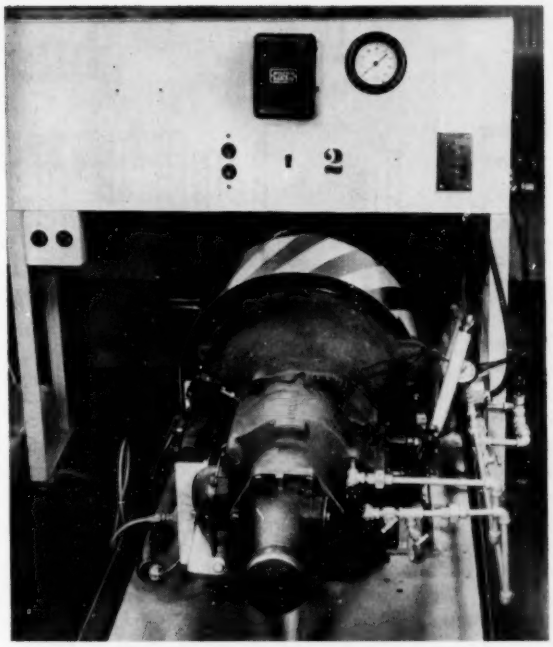


FIGURE 1, powerglide oxidation test stand.

been substituted for the L-4 procedure. This new test, popularly known as the Powerglide Oxidation test, is not subject to the objections enumerated for the L-4 test. In the Powerglide procedure, the fluid is tested in an environment and equipment representative of that encountered in actual service, the fluid is not diluted and the fluid is subjected to a severity level which is realistic in terms of that encountered under actual operation in modern transmissions.

Basically, the L-39 test consists of reversing the stator and stator sprag in a Chevrolet Powerglide transmission and driving the transmission with an electric motor at a speed of 1750 R.P.M. for 300 hours. Since the reversed stator and sprag subject the fluid to shear with the consequent generation of heat, the specified oil sump test temperature of 275°F. is maintained by means of an oil cooler and the proper temperature controls.

At the end of the test, the thrust washers are checked for weight loss, the suction screen is inspected for clogging, the clutch plates are examined for sticking and for transfer of clutch plate material and all parts are inspected for varnish and sludge. Figure I is a photograph of a Powerglide Oxidation Test Stand in operation at the Texaco Research center, and Figure 2 shows the overall transmission cleanliness ratings of runs on six fluids. The CRC Varnish and Sludge Rating system was used as a basis for assigning numerical values to the sludge and varnish deposits on the various parts. In this system, a rating of 100 represents absolute cleanliness.

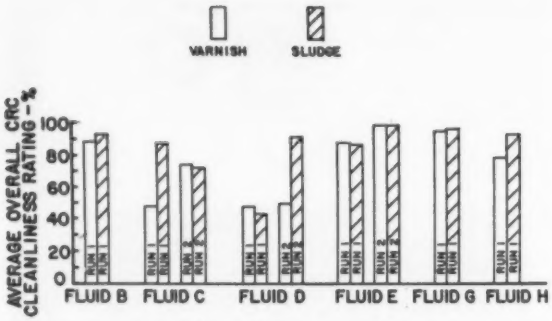


FIGURE 2, powerglide oxidation test (CRC L-39)

As previously indicated, the L-39 Powerglide Oxidation test is more severe than the Chevrolet L-4 test and satisfactory completion of the former by a fluid is intended to insure that the fluid possesses increased oxidation resistance. Under the new specification, a fluid which satisfactorily passes the Powerglide test and meets all the other requirements of the 1957 specification is designated as a qualified Type A, Suffix A product, the Suffix A indicating specifically that the requirements of the L-39 Powerglide Oxidation test have been met. Such fluids are assigned an "Armour Qualification Number" (AQ-ATF—A), the blank being filled with the specific number assigned to the fluid.

The fact that the Type A, and Type A, Suffix A, specifications have accomplished their purpose of assuring high-quality and trouble-free performance of automatic transmission fluids is indicated by Table II. This table lists, by type, all of the complaints pertaining to automatic transmission fluids received by Texaco from 1950 through the present. These complaints have been subdivided further into those relating to fluids supplied for factory-fill and into those originating from consumers who have registered complaints concerning fluids after they have been used in actual transmission service. The latter category includes those fluids which were delivered with the new vehicles in addition to those which were sold through service-station type outlets.

As can be noted, the total complaints in all categories numbers only 82, in spite of the fact that millions of gallons of Texamatic fluids have been sold and used during the time period covered. As shown, the largest total number of complaints involved contamination during handling and could not be attributed to fluid quality. The next largest number involved mechanical difficulties experienced with the fluids and, in almost all cases, resulted from either maladjustment of the transmission or operation under extremely severe service conditions for which neither the transmission nor the fluid was designed. Ten of the complaints involved contamination in service and usually could be traced to the introduction of water through leaky heat exchangers. Six of the complaints could be traced directly to the fact that the user had attempted to extend the drain period far beyond that recommended by the transmission manufacturer.

TABLE II

Automatic Transmission Fluid Complaints Received by Texaco (1950 To Date)

Type of Complaint	*Number of Complaints Factory-Fill*	*Resale or Service*	*Total*
Contamination of Fluid During Handling	28	12	40
Transmission Mechanical Difficulties	1	18	19
Contamination of Fluid During Service	0	10	10
Excessive Extension of Recommended Drain Period	0	6	6
Improper Handling by Customers	2	2	4
Objectionable Odor	2	0	2
Transmission Overfilled	0	1	1
Total	33	49	82

As can be seen, much effort has been expended in the past to insure that the users of automatic transmissions be supplied with fluids which have met high standards of quality and performance. This effort has been a joint one on the part of the automotive and petroleum industries. It is only through such continued close cooperation that the future advancement of both can be realized.

About the Author

W. H. Kite, Jr. *graduated from the University of Virginia in* 1943. *He then joined the U. S. Navy. After the war he taught at the University of Virginia School of Engineering for one year and at North Carolina State College in the department of diesel and internal combustion engines, for four years. While at N. C. State, he pursued his graduate studies and, in* 1951, *received an MS degree in diesel engineering. In* 1951 *Mr. Kite joined the research division of Texaco's research & technical department, where he was engaged in work in the fields of automotive combustion processes, abnormal combustion and fuels and lubricants research. In* 1955 *he was appointed to the staff of the manager of the technical services division, where, as staff automotive engineer he is engaged in all activities pertaining to the automotive field.*

Literature and Patent Abstracts

Characteristics

The Flow of Lubricating Greases

A. W. Sisko, *Ind. Eng. Chem.* 50, 1789-92, Dec. 1958.

After considering former suggestions, a relatively simple flow equation is given which fits experimental flow data for lubricating greases.

For the experiments five lubricating greases were chosen as are noted in Table 1. An ASTM viscometer was modified so that shear rates from 10 to 22,000 sec.$^{-1}$ could be obtained. Also the number of capillaries were increased and the range extended into that of a pipe viscometer. By this means it was possible to compare calculated values with those obtained by both the ASTM viscometer and a pipe viscometer.

Table 1

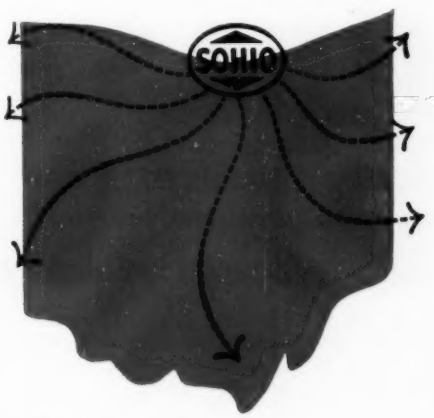
Grease	Thickener Type	%	Oil SUS at 100F.	Constants a	b	n
A	Ca FA	7.2	260	2.70	1940	0.139
B	Li Hydroxy Stearate	6.5	813	5.34	2300	0.196
C	Na Tallow	8.9	500	6.00	2380	0.224
D	Ca FA	8.5	1720	22.00	2000	0.175
E	Hydrophobic Silica	13.0	813	22.60	4650	0.054

The flow equation is:

F (flow rate) = a times shear rate + b times shear rate^{n} where a, b and n are constants which are also shown in Table 1 for the various lubricating greases tested.

Additives

"Additives Are Big Business" is the heading of a two page article in June 1, *C & E N*, which treats of additives for petroleum products.



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Lindert states that 75 million dollars are spent for addition agents in industrial lubes, road oils, waxes and greases. The consumption in lubricating greases is stated to be 60 million pounds and that in transmission and gear oils 72 million pounds of additives.

Effect of Molybdenum Disulfide Addition in Certain Lubricants

In view of the fact that molybdenum is being added to some lubricating greases, the investigations reported by Vineall and Taylor (*Scientific Lubrication* Vol. 11, No. 4, p. 32-35, 1959) will be of interest to our readers. The authors seek to answer certain criticisms of such usage.

First, they refute the statement that the presence of molybdenum disulfide catalyzes the oxidation of petroleum oils. Using a straight mineral oil, having a viscosity of 110 SUS at 100°F.: one portion had no addition; another contained 0.35 per cent colloidal MoS_2; and still another portion contained 1.0 percent of powdered MoS_2. After 24 hours air blowing at 200°C., the untreated oil had a viscosity of 153.3, the fraction with colloidal molybdenum disulfide had a viscosity of 148.5 (initial 107.8), and the portion with the powdered MoS_2 had a viscosity of 154.0.

Next, the statement is refuted that molybdenum disulfide will not lubricate brass and steel combinations and does, in fact, promote rather than prevent wear. For this purpose the friction and wear of combinations of the two metals were determined on a Timken lubricant and wear tester at lever arm loads of both two and fifteen pounds. While most of the tests were made first, with petrolatum and then with the same containing 50 per cent MoS_2, we are more concerned with tests where a Bentone grease with and without a 10 per cent addition of MoS_2 was continuously fed to the moving parts. In each case a brass block and a steel cup were used. With a fifteen-pound load applied for five minutes, a block wear of 507 mg. occurred with the untreated grease and of 23 mg. with that containing MoS_2. The coefficient of friction was 0.12 during the first test and 0.03 in the latter case. With a two-pound load applied for 30 minutes, the corresponding block wears were 195 mg. and 4 mg. and the coefficients of friction 0.09 and 0.04 respectively.

Finally, consideration is given to the statement that molybdenum disulfide oxidizes readily to an abrasive which promotes wear. While such oxidation only takes place above 750°F. a statement by these authors is of interest: "A test rig for friction experiments showed that although molybdic oxide was formed on heating to high temperatures,

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Moisture	0.5-1.2%	Iodine Value (Hanus)	20.0-35.0
Ash	0.1% Max.	Saponification Number	100.0-116.0
Melting Point	36.0°-44.0°C.	Penetration at 77°F. unworked	150-180 mm/10
Flash Point	540°F. approx.	worked	340-370 mm/10
		Color ASTM (Max.): 10%	4.5
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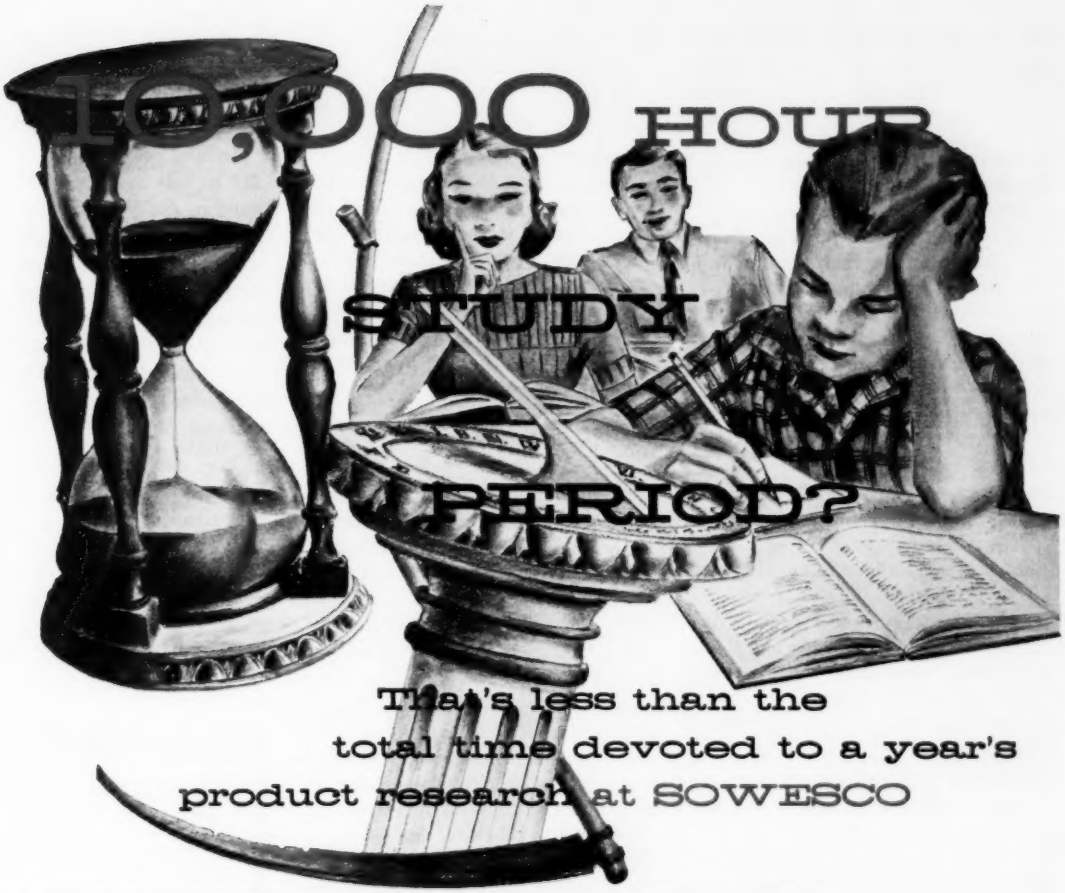

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the low friction of the sulfide operated as long as a layer remained in contact with the metal under the oxide."

Application

Chassis Lubrication

National Petroleum News-Fact Book Issue Mid-May 1959-60, p. 153 gives figures which seem to indicate that the consumption of chassis lubricants may not decrease this year. Some of the information of interest is tabulated below:

Year	*Average Number of Fittings per Car*	*Recommended Greasing Period-Miles*	*Private Registration Pass. Cars*	*Trucks*
1955	17.7	1,125	51,951,000	9,893,000
1958	10.7	1,215	56,700,000	10,660,000
1959	12.2	1,161	58,150,000	10,930,000

On the other hand, the July issue of *Popular Science* states that the first automobile to reach the market which requires no chassis lubrication is the DAF, a four passenger Dutch car.

Automotive Transmissions

The National Petroleum News-Fact Book p. 153, also gives information on automotive transmissions in use. Some such figures follow:

AUTOMOTIVE TRANSMISSIONS IN USE

Year	*No. in Use*	*% of Car Registration*	*Capacity in Quarts*
1955	20,073,000	39.6	10.1
1958	30,090,000	52.8	8.9
1959	33,195,000	56.5	8.9

Fuels and Lubricants for the Trans-Antarctic Expedition

D. L. Pratt, G. M. Christie, E. F. Coxon and J. R. Lodwick. J. *Inst. Petroleum*, 45, 19-41, Feb. 1959.

Comments of the authors relative to the lubricating grease and the gear oil used on this expedition are of interest. The equipment to be serviced consisted of seventeen tractors of four different makes.

The lubricating grease was used primarily for the lubrication of track rollers and for this purpose MIL - G - 10924 low - temperature product was chosen. Care was taken that all rollers and other grease-packed bearings were filled with this type of product before shipment. While planning the expedition, consideration was given to a water pump lubricant because the engine temperatures would reach 180°F. However, field trials in Norway demonstrated that a product meeting specification MIL-G-10924 was satisfactory for water pump lubrication.

It was stated that "The grease did its job well, especially with regard to the Sno-Cat rollers." However, mention was made that three outer pontoon bearings did break up but that these bearings were pre-packed and perhaps overloaded in use. Also, one pre-packed input-drive-shaft transfer gear box bearing failed, this being one out of 24 such bearings on Sno-Cats.

The gear oil supplied for Sno-Cat and Weasels was one meeting specification MIL-L-10324 but for Ferguson tractors an oil of similar viscosity and cold test but with a difference in the load-carrying additive was provided. In this case the phosphorus content of the compounded oil was 0.080 per cent and

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the zinc content 0.083 per cent. No gear box defects due to lubrication were experienced.

Choosing Lubricants for Heat and Cold

Alden Crankshaw. *Machine Design* 31,101-03, February 5, 1959.

Broad temperature ranges are considered with no detailed specifications of recommended products. Over a range of −65 to 130°F. it is stated that a number of greases will perform well if "properly applied." It is cautioned that an extremely thin film must be applied on friction surfaces and that too liberal application will cause the mechanism to "freeze" at extremely low temperatures.

The next temperature range considered is 0 to 350°F. and the author states that under these conditions "temperature can be relegated to the background and choice based on bearing type, speed, load and-even personal preference."

Finally, solids are suggested for very hot applications.

Theory

Viscoelasticity in Lubrication

G. J. O'Donnell. *Mech. Eng.* 81, 63-65, Feb. 1959.

Some of the comments of the author are of interest because our readers will be faced with this term to a greater extent in the future. This will probably be especially true in the case of gear lubricants. A couple of quotes will suffice for the present.

"The phenomenon (performance of spur gears at high speeds) is that of decreasing load-carrying capacity with increasing speed up to a critical speed, and then increased load-carrying capacity with increasing speed." "Depending on the rate of application of a shear stress to a liquid, the shear response could be that of a normal viscous liquid or of an elastic solid. Or, if it responded as a combination of both, it would be what is known as a 'viscoelastic' substance."

Composition

Clay-Thickened Lubricants

An improvement in the worked stability of lubricating greases thickened with organophilic bentonite consists of use of a dispersant which is either a mixture of acetonitrile and acetone or a mixture of propionitrile and acetone. These compositions are described by Stratton in U. S. Patent 2,879,229, assigned to Phillips Petroleum company.

Table I shows first, the variation in composition of the dispersant and next, the penetrations of the resulting lubricating greases after being subjected to 60 double strokes and 10,000 double strokes in a grease worker. While grease No. 4 shows the least difference between the two values it should be kept in mind that the total dispersant varies in the different compositions.

Table I

Grease No.	1	2	3	4	5	6
Composition Wt. Per cent						
"Bentone 34"	5.50	5.50	5.50	5.50	5.50	5.50
Paraffinic SAE 20 Oil	93.50	93.25	93.00	92.75	92.62	92.50
Acetonitrile	1.00	0.75	0.50	0.25	0.125	0.00
Acetone	0.00	0.50	1.00	1.50	1.75	2.00
ASTM Penetrations						
60—strokes	302	302	307	314	339	386
10,000—strokes	331	331	331	336	362	411

The mixtures cited above were milled in a Charlotte mill with a T-grooved rotor and stator at 0.003 inch clearance.

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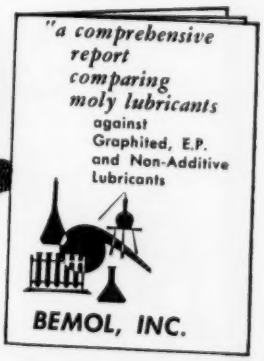

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. . . How Greases Behave at -65°F is examined by chemist in International Lubricant's New Research Laboratory

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Soap-Salt Complex Thickened Lubricating Greases

Lubricating greases having high dropping points and excellent load carrying ability are formed if the thickener consists of soap-salt complexes comprising a metal salt of acetic acid, a metal salt of cyclodiene dicarboxylic acids or dienophilic acid derivatives thereof and a metal soap of conventional grease-forming high molecular weight fatty acids.

By cross-polymerizing dimers of monocyclodiene monocarboxylic acids with maleic anhydride, a suitable tricarboxylic acid or anhydride thereof is obtained.

Morway and Wiese, who described these lubricants in U. S. Patent 2,880,174, assigned to Esso Research and Engineering company, find that a mol ratio of acetic acid to the other acids is preferably 7:1 to 12:1 and the ratio of the cyclodiene dicarboxylic acids to the high molecular weight fatty acids is preferably 0.5:1 to 2:1.

Ingredients for a typical formulation consisted of 12 per cent glacial acetic acid, 3 per cent dicyclopentadiene dicarboxylic acid, 3 per cent stearic acid, 9.7 per cent hydrated lime, 0.5 per cent phenyl alpha naphthylamine, and 71.8 per cent of a lubricating oil having a viscosity of 55 SUS at 210°F. In manufacturing the lubricant all of the oil, the dicarboxylic acid, the stearic acid and the lime were charged to a fire heated kettle where they were mixed and warmed to 135°F. The acetic acid was then added and heating was continued to 500°F. The mass was then cooled to 275°F. and the oxidation inhibitor was added before further cooling to 180°F. and Gaulin homogenizing at 6000 psi.

The resulting lubricating grease had a dropping point of 500+°F. an unworked penetration of 302, and worked penetrations of 320 after 60 strokes and 345 after 10,000 strokes. This product had a lubricating life of over 1900 hours in a bearing operating at 10,000 rpm and 250°F. Also it passed a Timken load test of 40 pounds.

Industry News

Lab Survey on Film Lubricants Available

A twenty page brochure has just been released by Bemol, Inc., covering the comparative values of moly, synthetic graphite, natural graphite, chemical EP, and non-additive lubricants.

Over fifty composite charts in this comprehensive survey compare moly against the other film lubricants on the Falex, Timken, Shell four-ball, and kinetic friction testing machines. The study examins the lubricants in relation to their frictional properties, wear reducing abilities, film endurance, load capacities, and temperature reducing abilities. The results show the superiority of moly in boundry, oscillating, and slow speed operation; (and the effectiveness of moly under high speed testing.)

The report covers the addition of moly and the other solid and chemical additives to greases, oils, synthetics, and bonded coatings. Copies of the report are available from Bemol, Inc., 131 State St., Boston. Bemol lubricant engineers are located in Chicago, Cleveland, Youngstown, Fort Worth, and Spokane.

Low-Cost Greasing Unit

An inexpensive, compact greasing unit has recently been announced by the Battenfeld Grease & Oil Corporation. Outstanding features of the new unit, designated "Jiffy-Luber" are (Gun): lightweight steel construction, versatile, will develop 1500 pounds pressure psi, will "open" the ball check in most any grease fitting. (Bracon grease tube): sturdy, tough plastic type tube filled with white multipurpose lithium grease.

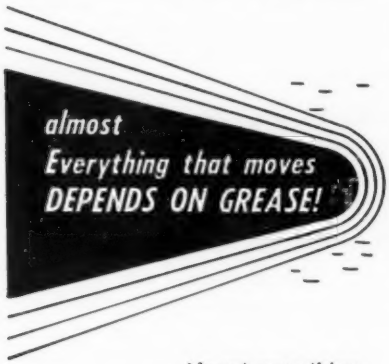
The Jiffy Luber units may be used by do-it-yourselfers in the home workshop or by professionals for special applications. These new units serve equally well for special promotion items.

For further details, contact Mr. Robert E. Duroche, director of publicity.

Fatty Acid Council Produces Book

Industrial Fatty Acids and Their Applications has been offered by the Reinhold Publishing corporation, 430 Park avenue, New York 22. Edited by E. Scott Pattison, manager of the Fatty Acid Producer's Council, the book deals with chemistry, processing and production of fatty acids, with emphasis on practical technology and the chemistry involved with it.

The coverage also includes an up-to-date review of the applications of fatty acids in metallic soaps and greases, in a chapter written by E. F. Wagner of Witco Chemical company.



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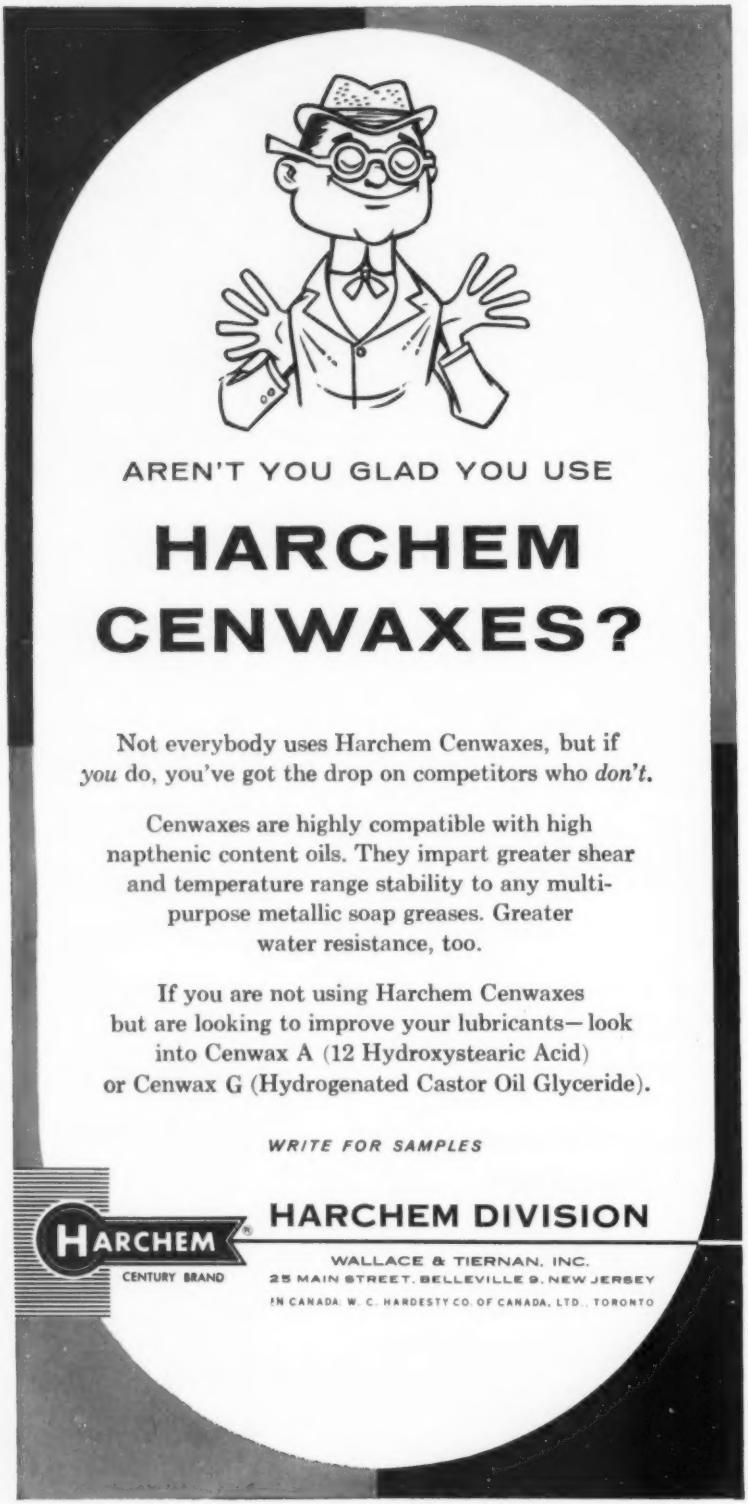
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Alpha-Molykote Modifies Testing Machine

The Alpha Lubricant-Friction-Wear testing machine (Model LFW-1) has been modified to add two new testing speeds, it was announced by the Alpha-Molykote corporation, Stamford, Conn.

The new speeds serve to increase the original multi-speed operating range of the machine in two directions. Originally manufactured with testing speeds of 35, 72 and 120 rpm, the Model LFW-1 is now available with a 12½ rpm speed on the low side and a 200 rpm speed on the high side of the range. In friction and wear testing, velocity is the key variable and the newly-increased testing speed range of the Model LFW-1 makes possible more complete studies of this variable.

For more complete details, write the Alpha-Molykote corporation, 65 Harvard Avenue, Stamford, Conn.

BP Builds New Blending Plant on Yarra River

A new lubricating oil blending plant on the Yarra river, at Spotswood, Melbourne, has been built by BP Australia, Limited. A total of 180 different grades and types of lubricants will be blended and packed at the new plant for BP's Australian and New Zealand markets.

Facts for Industry Survey Results Announced

Sales of lubricating oils and greases as reported by approximately 150 companies totaled 53,552,835 barrels in 1958, according to a "Facts for Industry" survey prepared by the Bureau of the Census under sponsorship of the Lubrication Committee of the Marketing Division, American Petroleum Institute.

A breakdown of the total sales shows that: 22 percent was sold for direct shipment to destinations outside the continental United States;

2.8 percent was sold to the federal government; and the remainder of about 75 percent went to customers located in 48 states and the District of Columbia.

The sales figure of 53,552,835 barrels is composed of 49,824,975 barrels (42 gallons to the barrel) of oils and 3,727,860 barrels (315 pounds to the barrel) of greases.

Copies of the 11-page survey entitled "Sales of Lubricating Oil and Greases, by States, Types, and Company Classification, 1958" are available at ten cents each from the Bureau of the Census, Washington 25, D. C. It was prepared by the bureau's Industry Division, Chemicals.

The national survey was the fourth of its kind. Others were conducted in 1947, 1951, and 1956. Companies canvassed were specially selected so as to reflect estimates of lubricating oil and grease consumption by states. In order to keep the number of reporting firms as small as possible, the canvass was limited to all primary producers and to those relatively large compounders and marketers who distributed lubricating oils and greases on an interstate basis.

Sales to all companies other than prime producers and specified marketers and compounders were excluded from the survey to avoid duplication by inclusion of intercompany sales. In addition, other reporting provisions were made so that no significant sales duplications would occur. The survey statistics therefore exclude companies primarily engaged in distributing their products intrastate.

Chairman of the Marketing Division's Lubrication Committee, which sponsored the survey, is H. P. Ferguson, manager of wholesale and subsidiary sales, the Standard Oil Co. (Ohio), Cleveland, Ohio. Chairman of the Lubrication Committee's statistics subcommittee, which worked with the bureau in developing the survey, is P. W. Zumbrook, Sinclair Refining Co., New York.

ASTM Committee Conducts Poll

ASTM Sectional Committee Z11 on Petroleum Products and Lubricants is currently polling members on 25 recommendations which would cover the approval of seven new American standards and eighteen revised American standards.

Two of these American standards deal with lubricating greases . . . D 1261–55, Method of Test for Effect of Grease on Copper, and D 1262–55, Method of Test for Lead in New and Used Greases. A revised American standard, D 128–59; Z11.16, is Methods of Analysis of Lubricating Grease.

Votes will be canvassed on September 18, whereupon the results will be reported to the American Standards Association.

Engineer Discusses Spray Lubrication

A technical paper, "Modern Techniques for Spray Lubricating Industrial Gearing," has been pre-

If it rolls on an axle or turns in a
bearing or rides on a shaft if it slides
in a groove or moves on a pivot if
it bores or cuts or transmits pressure
one of Sinclair's 500 specialized lubricants
is designed to make it work better. For answers
to your lubrication problems, write today to
SINCLAIR REFINING COMPANY Sinclair
Technical Service Division, 600 Fifth Ave., N.Y. 20 N.Y.

pared by E. J. Gesdorf, senior application engineer of the Farval corporation, manufacturers of centralized lubricating equipment. Interesting application photographs and cutaway views of spray equipment are contained in the treatise. Included is a discussion on historical development of spray lubrication, followed by information on problems encountered and how these problems were solved.

Copies are obtainable from the Farval corporation, 3300 East 80th street, Cleveland 4, Ohio.

Foote Breaks Ground For Research Laboratory

Foote Mineral company broke ground recently for its new $2.2 million research and engineering building near Exton, Pa. L. G. Bliss, Foote president, turned the first spadeful of earth at informal ceremonies.

The modern multi-wing building will be the latest addition to the Company's 53-acre research and engineering center which has been under development since 1957. It will house the firm's research and development department, central engineering department and will provide administrative offices for the production department. It is designed to accommodate a staff of about 175 scientists and engineers. Foote's research and development staff has doubled within the last two years and continued expansion is anticipated. The new facilities will supplant the now overcrowded buildings in Berwyn, Pa., and at the company's Exton plant location.

Chek-Chart Announces Publication of a New Tractor Lubrication Guide

The Chek-Chart corporation has just announced the publication of a new Tractor Lubrication Guide.

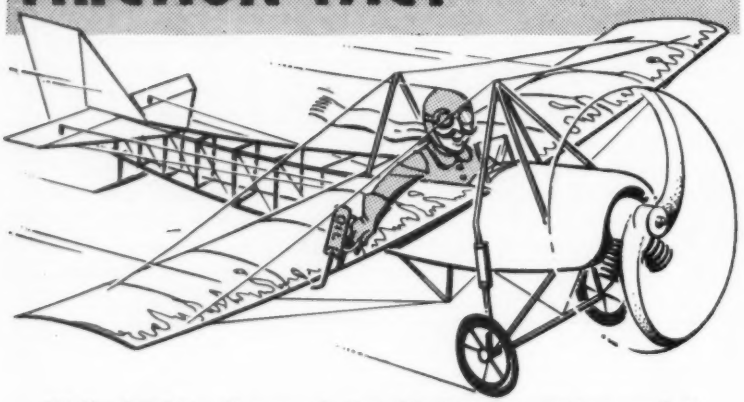
The Guide contains 64 fact-filled pages of manufacturer-approved tractor lubrication information on all popular makes and models of farm tractors. Each of the tractor lubrication diagrams clearly indicate the exact location of every point of lubrication, with specific instructions on the interval at which these points are to be serviced and the exact lubricant to be used.

All new in this edition of the *Tractor Guide* are two tabular pages of motor oil and gear lubricant recommendations and capacities for crankcase, transmission, and final drive units. Five pages of service instructions provide complete service information on all phases of tractor lubrication.

This new guide contains all the lubrication information needed to keep tractors operating in an efficient, economical, and trouble-free manner.

Complete information and prices on request.

FRICTION & FACT From Les' ALMANAC



Wilford Write discovered that his flying machine worked much better when he lubricated the leading edges of his wings with a light oil, thus enabling the craft to slide through the air with a minimum of friction resistance.

☐ *True* ☐ *False*

The answer of course is "False" but for any problem of friction involving machinery, Cato has the "True" answer.

Your customers' lubrication problems are solved when they use Wanda products or our custom-made oils and greases made to your specifications. They'll get outstanding results from any product in our complete line which includes lithflex, emerald gun, pressure gun, viscid lube, heavy duty oil, brake fluid, anti-freeze, and industrial oils. Customer satisfaction means FAST TURNOVER and BIG PROFITS for you.

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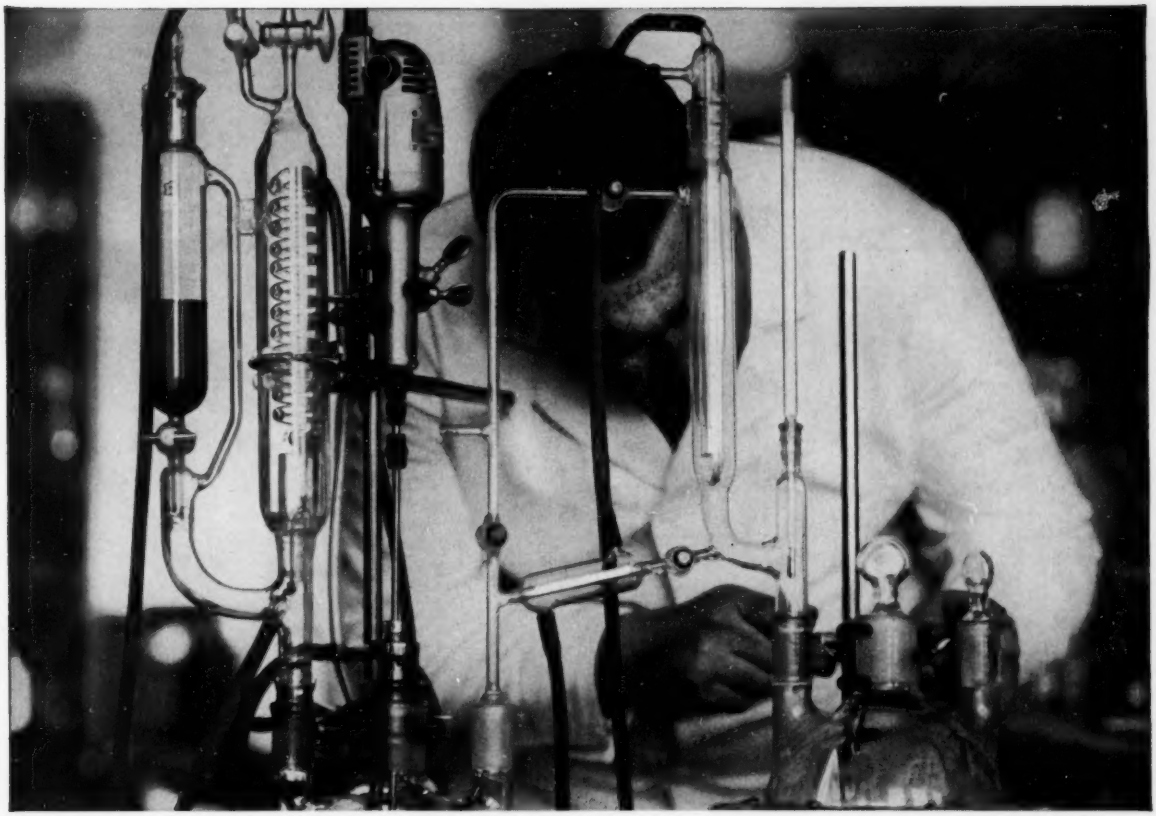


How organic chemists put lithium to work

Recent interest in organolithium compounds owes much to the fact that these compounds are soluble in hydrocarbons. The reactions of the organolithium compounds resemble those of organomagnesium compounds, yet have distinct advantages. In solution, lithium compounds exhibit a degree of reactivity intermediate between alkali and magnesium reagents.

Where it is necessary to use ether solvents, it is found that organosodium compounds decompose most ethers too rapidly. The organomagnesium compounds have too slow a reaction rate to be useful. With organolithium compounds the desired reaction can be completed before the ether is substantially attacked.

To produce intermediates for further reaction, certain ethylenic and aromatic systems add lithium and other alkali metals to give metallic derivatives. Lithium appears to react more readily than sodium or potassium and sometimes follows a different course of reaction.

Lithium metal and lithium alkyls seem to have the ability to direct the course of a polymerization. Isoprene has been polymerized to a product containing over 93% cis-1,4 addition product. Such polymers are considered to be the nearest approach to natural rubber. This stereospecific behavior of lithium catalysts may be useful in other organic reactions.

Reduction by means of alkali metals can be accomplished by using sodium in high-boiling solvents and in liquid ammonia. Recently it has been reported that the use of lithium often gives better yields. The versatility of lithium as a reducing agent in ethylenic and aromatic compounds is shown by the selective reduction of the carbon-carbon double bond of a conjugated ethylenic ketone using lithium in liquid ammonia. A contrasting example is the selective reduction of the carbonyl group of an unsaturated ketone using lithium aluminum hydride.

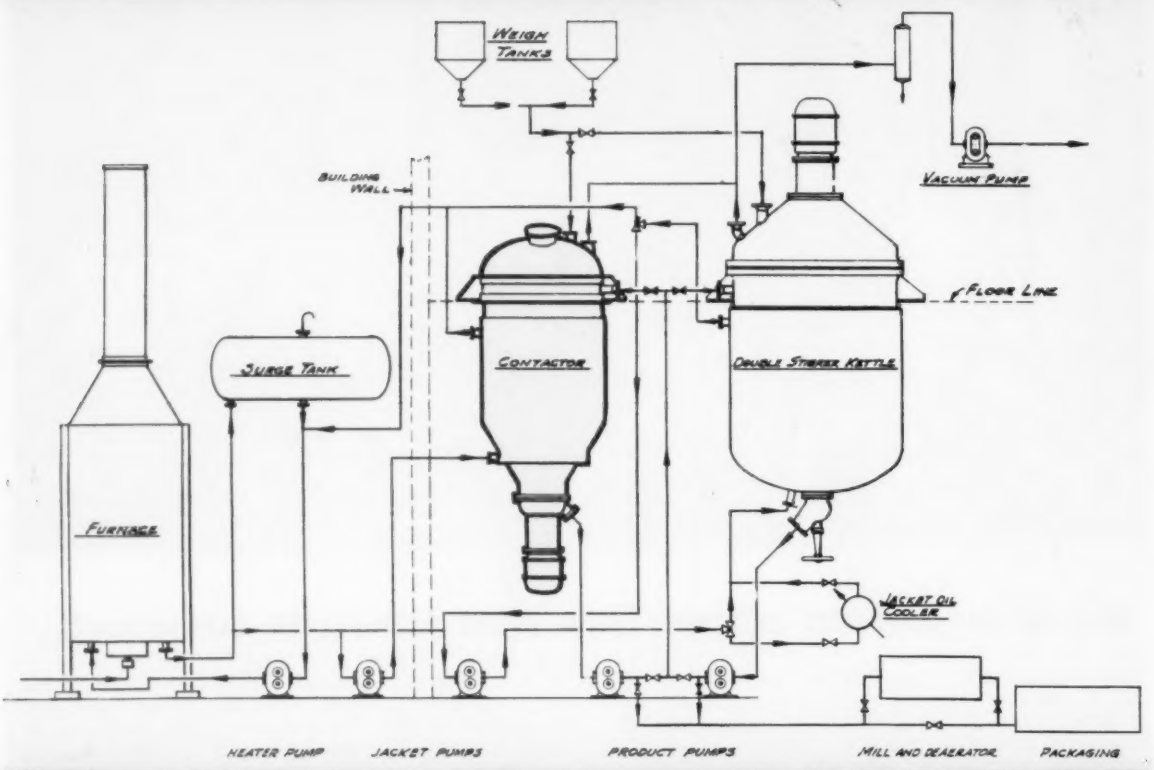
But this is only the beginning. Though the information on lithium in organics is relatively limited, its vast potential in this field is already well established. We'll be glad to share this information with you if it can help you in any way with your specific organic problem. Address letterhead request to Technical Literature Department, Foote Mineral Co., 402 Eighteen W. Chelten Bldg., Phila. 44, Pa.

FOOTE MINERAL COMPANY

LITHIUM CHEMICALS, METAL, MINERALS • STRONTIUM CHEMICALS • WELDING GRADE • FERRO ALLOYS • ELECTROLYTIC MANGANESE METAL • STEEL ADDITIVES • COMMERCIAL MINERALS AND ORES • ZIRCONIUM, TITANIUM, HAFNIUM (IODIDE PROCESS)

The STRATCO contactor

Reg. U. S. Pat. Off.



and How It Simplifies Grease Making

The Stratco contactor is a highly efficient mixing device and when combined with the heating and cooling system shown above provides extremely close control of reaction temperature. With intimate contact between reactants and controlled temperature, very short batch time cycles are required.

Compared with other systems, Stratco grease plants produce more uniform greases with less soap and require less laboratory control.

A complete Stratco plant layout is illustrated above. Equipment is adaptable to modernization programs as well as new installations. Specific equipment recommendations made without obligation.

STRATFORD ENGINEERING Corporation

612 West 47th St. — *PETROLEUM REFINING ENGINEERS* — Kansas City 12, Mo.